APPLIED Spectroscopy

1961
Vol. 15 No. 6

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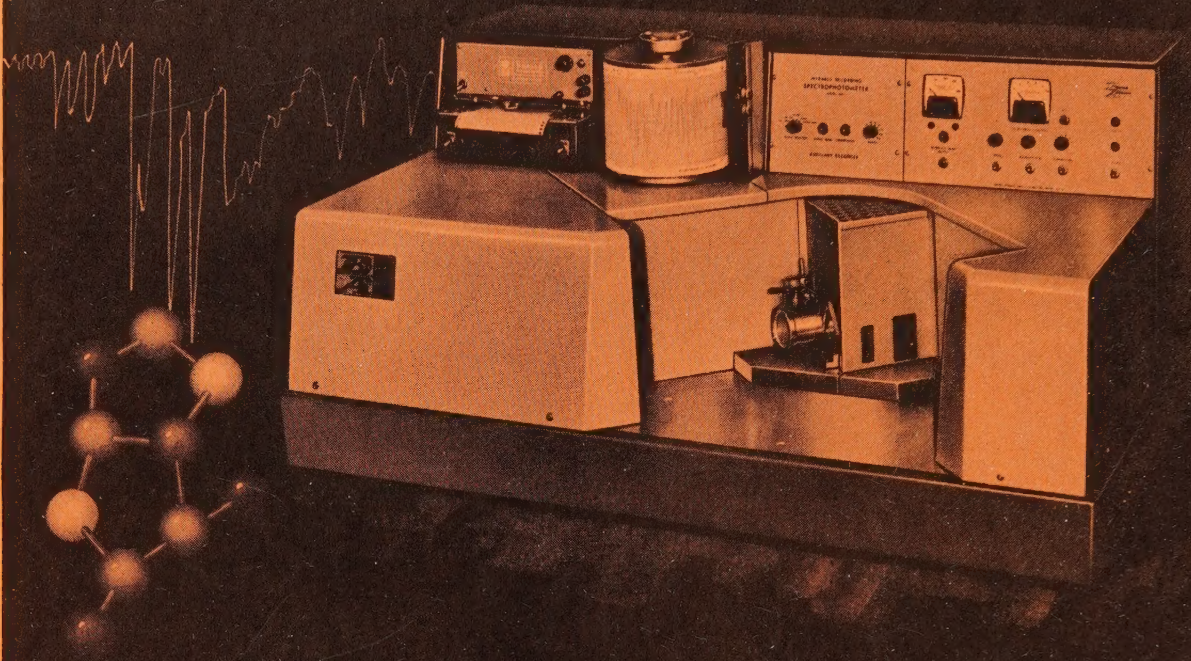

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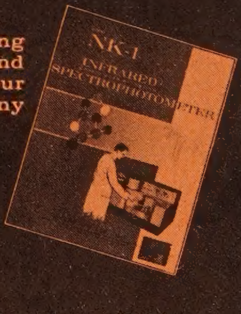

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Contributions covering the theory and practice of absorption spectroscopy (x-ray, ultraviolet, visible, infrared and microwave), emission spectroscopy (arc and spark, flame and fluorescence), Raman spectroscopy, diffraction, mass spectroscopy and nuclear magnetic resonance spectroscopy are solicited. They may be in the form of *papers, notes,* or *laboratory suggestions.* Contributors need not be members of the Society. Contributions should be sent in *duplicate* to

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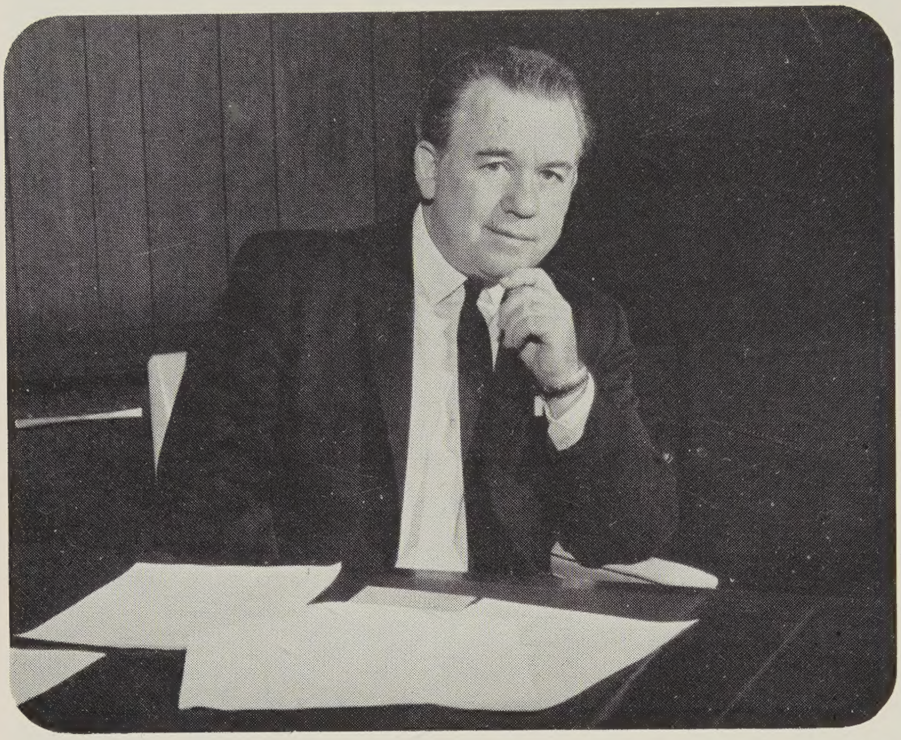

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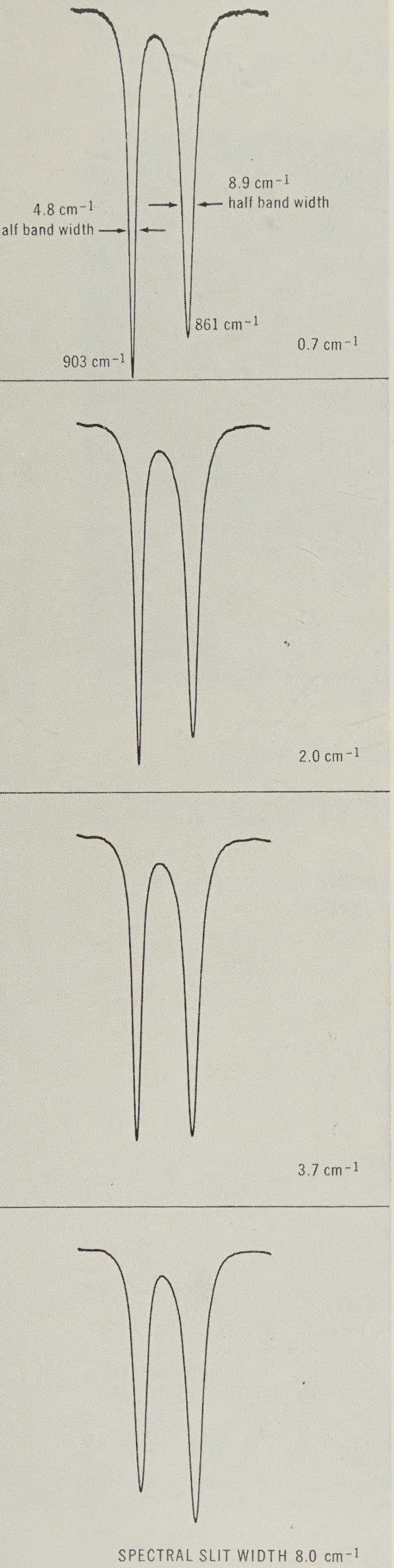

Left, spectrum of a 0.21 mm. thickness sample of spectro-grade cyclohexane demonstrates the importance of high resolution for routine *quantitative analysis.*

Right, the C-H stretching band of methane illustrates the increased research *data obtainable with high resolution spectroscopy.*

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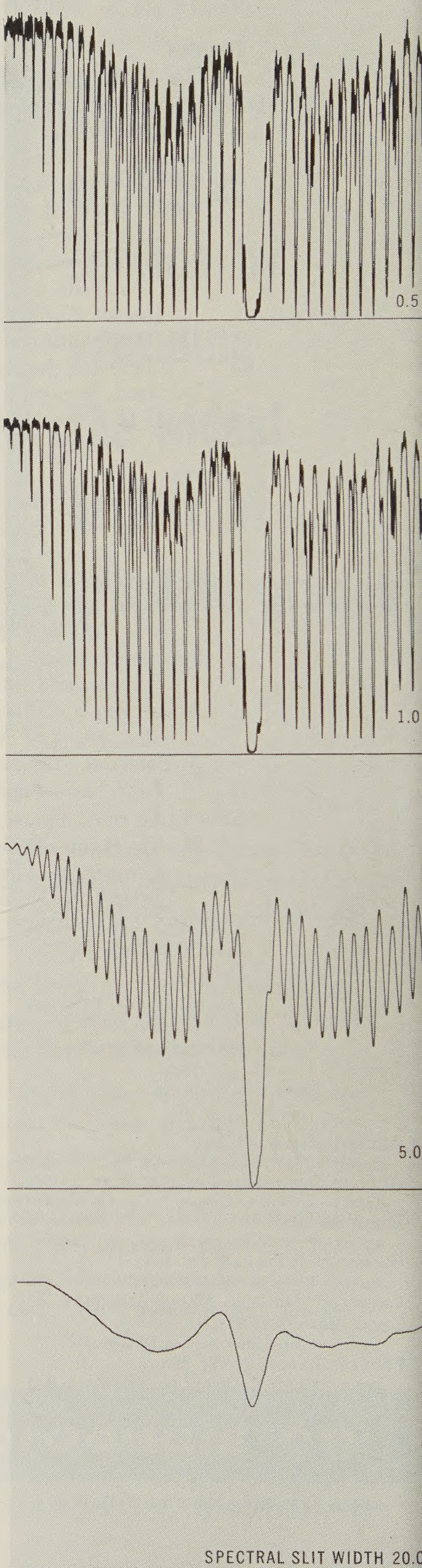


What's New?

Editor—Rodger W. Loofbourow

[Contributions for this column should be sent to its Editor, Mr. Rodger W. Loofbourow, Crobaugh Laboratories, 3800 Perkins Avenue, Cleveland 14, Ohio.]

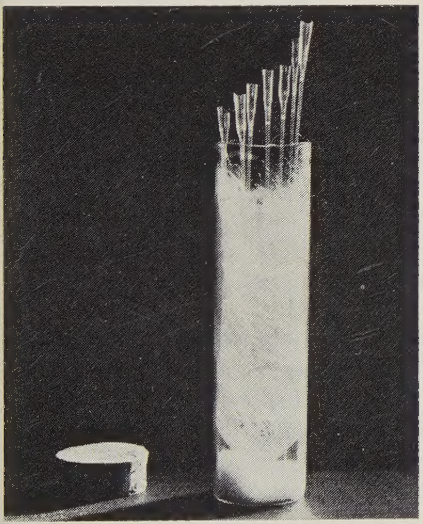
Applied Research Laboratories is offering a *light scattering* instrument, the Photo/Gonio/Diffusometer (PGD). This instrument is manufactured in France by ARL's subsidiary organization, Societe Francaise d'Instruments de Controle et d'Analysis.

Application of this instrument is in the analysis of suspensions, emulsions and products composed of giant molecules by means of light diffusion. Provision is made for studies at controlled temperatures over a range from low temperatures to a maximum of 150°C.

——— λ ———

Controlled wall thickness *glass or quartz capillaries* are available from the Caine Scientific Sales Company, 4120 West Lawrence Ave., Chicago 30, Ill. for x-ray diffraction camera work.

The thin walled capillaries are available in a choice of diameters from 0.2 to 2.0 mm. Each are 75 mm in length and have a wall thickness of 0.01 mm. They are sealed at one end and terminated with a funnel at the opposite end to facilitate filling.



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The *Periodical Monitor* provides the engineer with immediate reference to hundreds of articles on electronics and instrumentation each month. It is an abstract journal published by The Periodical Monitor and Abstract Service, 15 N. Euclid Ave., Pasadena, Calif.

Coleman Instruments, Maywood, Illinois, has developed a *spectrophotometer* designed for the automatic analysis of multiple samples for a single component.

The Chem-Matic Model 34 is capable of analyzing up to thirty samples within half an hour with results printed out on tape in any desired unit. Provision is made for the metered addition of reagents to dilute, remove interfering substances and develop color in the sample. Accuracies of better than 5% are claimed for most methods.

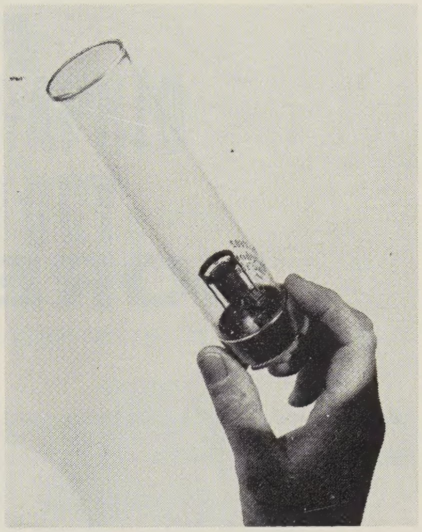
——— λ ———

The American Petroleum Institute Research Project 44 and the Manufacturing Chemists' Association Research Project were moved from Carnegie Institute of Technology, Pittsburgh 13, Pennsylvania to the A. and M. College of Texas, College Station, Texas on April 1, 1961. Both Projects issue standard *reference spectrograms* in each of five categories of spectroscopy—infrared, ultraviolet, Raman, mass, and nuclear magnetic resonance. The two Research Projects now constitute the Chemical Thermodynamic Properties Center in the Department of Chemistry of the College. Dr. Bruno J. Zwolinski, Professor of Chemistry, is the Director of the Center and the two Projects. Dr. Alfred Danti, Associate Professor, is the Assistant Director and is in charge of the Spectral Program. Spectra issued by the two Projects are contributed by responsible investigators. Contributions of spectra should be addressed to the Director or Assistant Director at the following address: Chemical Thermodynamic Properties Center, Department of Chemistry, A. and M. College of Texas, College Station, Texas.

——— λ ———

A new line of *hollow-cathode discharge tubes* for use in atomic absorption spectroscopy is now available from the Westinghouse Electronic Tube Division, Elmira, New York.

Tubes are available for analysis of materials containing iron, copper, nickel, zinc, manganese, magnesium, calcium, and other elements. The tubes feature low starting voltages, low operating voltage and stable current. The output spectral lines are narrow and steady after a short warm-up period. Special tubes with variations of cathode material, gas fill, and output window are available on request.



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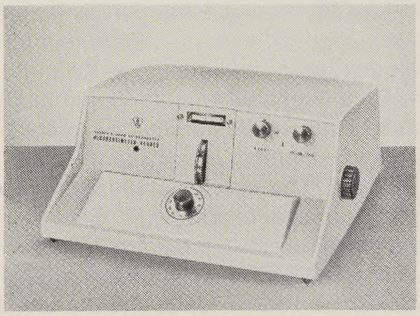
A new *Microdosimeter Reader*, designed for the measurement of X-ray, Gamma, or high energy Electron radiation has been developed by Bausch & Lomb, Incorporated, Rochester, New York.

The range of the new unit is 10 to 10,000 rads, with an accuracy of ±4%. The reader records the changes in fluorescence which take place in the tiny (1mm x 6mm) Microdosimeter Rods as the result of high energy radiation exposure. Complete measurement can be made within 15 seconds.



A *Cathode-Ray Tube Display Console* designed to be used with an electron beam micro-probe analyzer is offered by Elcar, Inc., Falls Church, Virginia.

Four cathode-ray tubes are provided to permit the simultaneous display of various characteristics of the target. The tubes have a common raster but are individually adjustable with respect to brightness, contrast, focus, position and image size. Provision is made for mounting an oscilloscope camera in front of each display tube.

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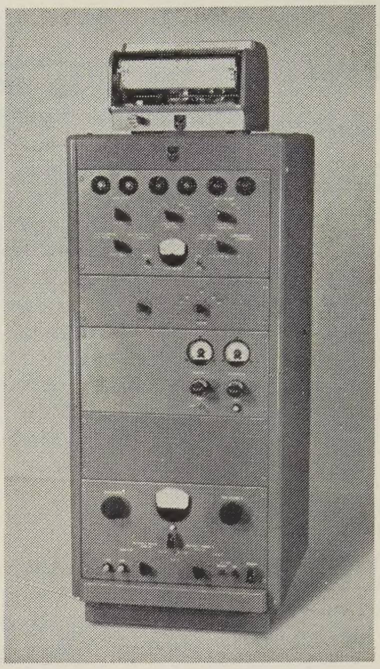

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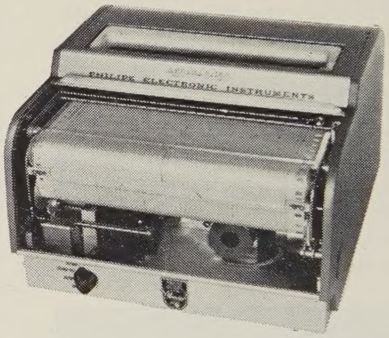

The many panel functions include: LINEAR and LOG ratemeter—the log rate meter automatically selects time constant depending on counting rate; INPUT SELECTOR which allows front panel selection of two pre-connected standard detectors, a Norelco dual detector, or an internal calibration signal; DECADE PRESENTATION will indicate fixed count to ten million counts, or fixed time to ten thousand seconds in a visual decimal display; PULSE HEIGHT ANALYZER with integral or differential scanning of 0-50 volts Baseline with Window Width adjustable within the voltage range. Pulse height analysis Window and Baseline controls read directly in volts and tenths of volts; DUAL HIGH VOLTAGE supplies stabilized direct current simultaneously for two detectors.

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The Spectral Line

Editor—S. Richard Wiley

[News about members and matters of interest to spectroscopists may be sent to the Editor, S. R. Wiley, 1239 Owen Brown Rd., Ellicott City, Md.]

William A. Wildhack has been appointed Associate Director of the National Bureau of Standards. He will be responsible for the coordination of the Bureau's measurement services to science, industry, other agencies of the Federal government, and the states. These services include consultation and research on special problems of measurement, and calibrations and tests performed on instruments, equipment, and materials, and the distribution of standard samples and reference materials.

Mr. Wildhack joined the Bureau staff as a physicist in the Aeronautical Instruments Section in 1935. He was chief of the Missile Instrumentation Section from 1948 to 1950. In that year, he was appointed Chief of the Office of Basic Instrumentation and has been serving as Special Assistant to the Director since 1960. Mr. Wildhack pioneered the practical development of the "Peek-a-Boo" retrieval system and its use in the instrumentation reference center.

After receiving a B.S. in electrical engineering, he obtained a M.S. in physics from the University of Colorado. He taught physics and mathematics at the University, Boise Junior College, and George Washington University. The Royal Swedish Academy of Engineering Sciences awarded him the silver medal in 1949 and he was named as the first Fellow of the Instrument Society of America in 1959.

——— λ ———

Dr. Mack Harvey is now associated with the El Paso Gas Company in charge of the analytical laboratory facilities at El Paso, Texas. He was formerly associated with the W. R. Grace Washington Research Center where he was in charge of the Absorption Spectroscopy Laboratory.

——— λ ———

Dr. Leopold May has been promoted to Associate Professor of Chemistry at The Catholic University of America. He is also serving as Managing Editor of Applied Spectroscopy.

(Continued on page 13A)

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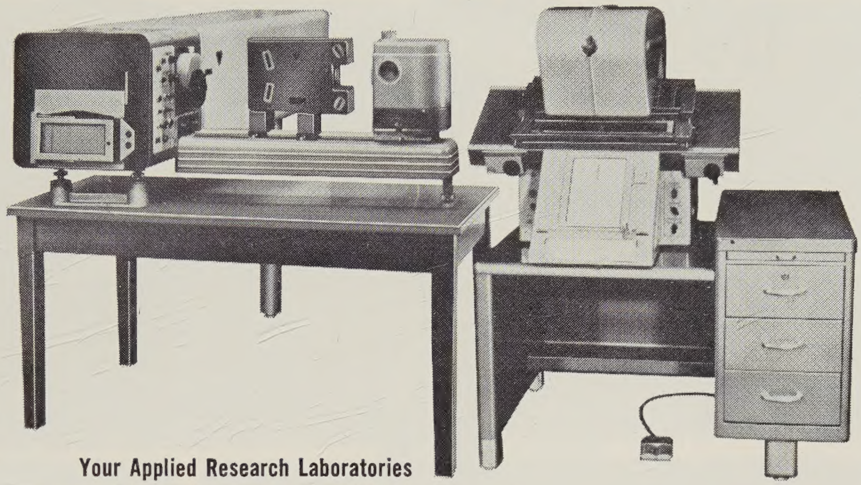

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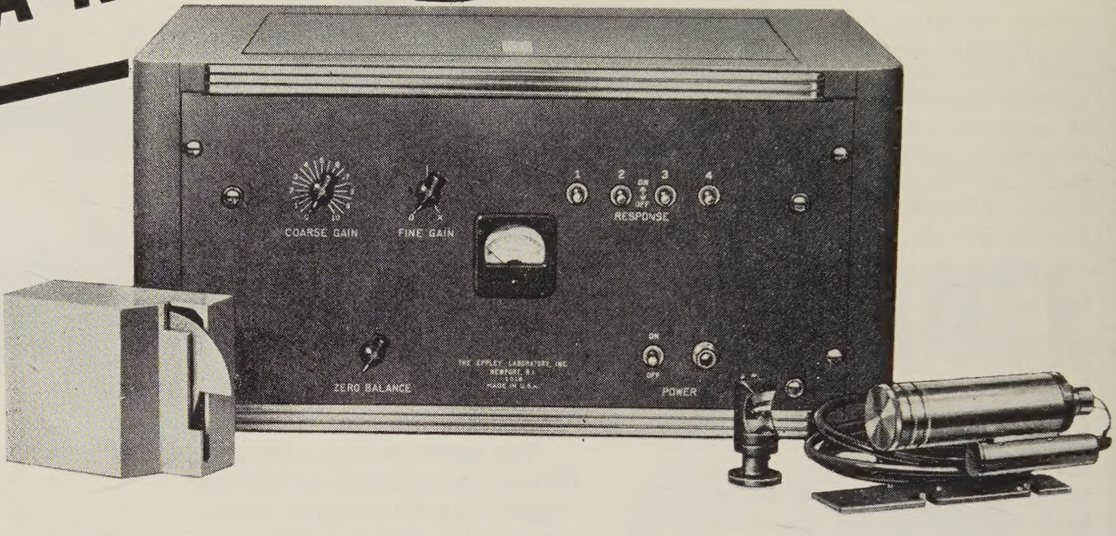

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*Fundamental and experimental aspects of this detector are discussed in the following publications: Rev. Sci. Inst. **18**, 347 and 357 ('47); ibid. **20**, 816 ('49); Proc. IRE **40**, 1161 ('52).

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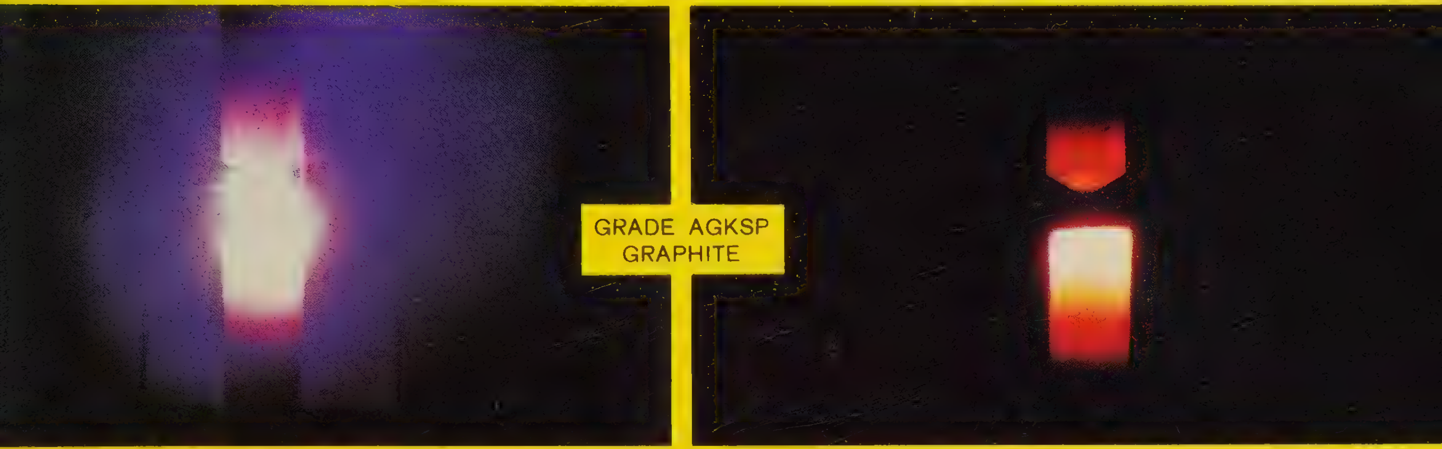
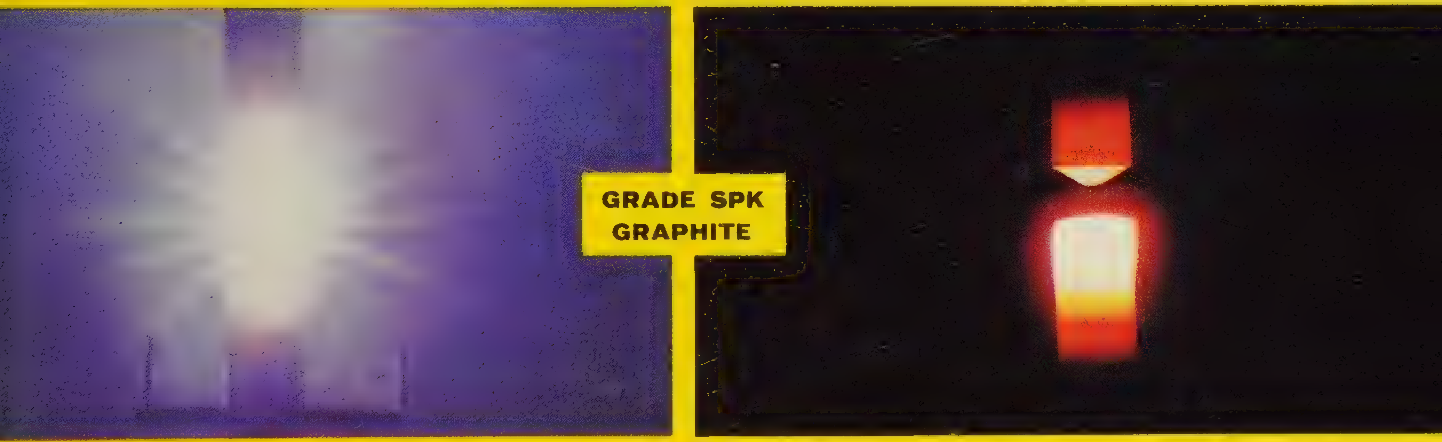
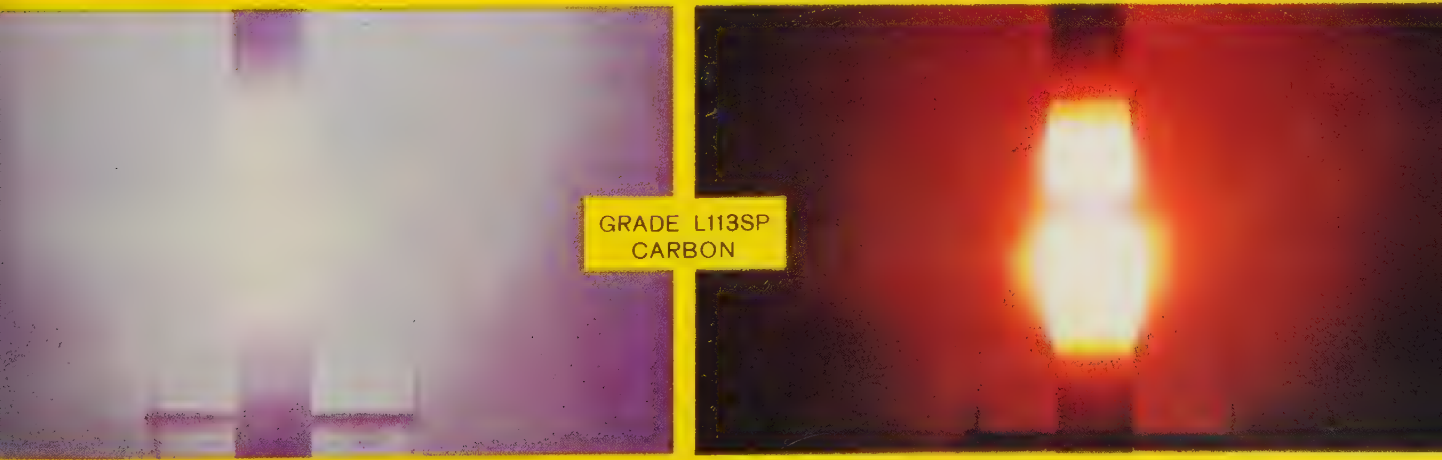




de SPK Graphite's excellent thermal properties, as illused, result in improved *stability* and *reproducibility*.

With Grade SPK Graphite, the heat is concentrated in the sample zone, thus insuring *minimum background effect*.



Although some applications require the extremely high temperatures of the carbon arc (above), excessive heat outside the sample zone contributes materially to increased background effect.

Turn to The Following Page for Information on Physical Properties of These 3 Grades

NATIONAL SPK GRAPHITE

TRADE-MARK

provides the high density, uniformity, and purity essential to

CONSISTENT, DEPENDABLE REPRODUCIBILITY

GRADE AGKSP GRAPHITE →

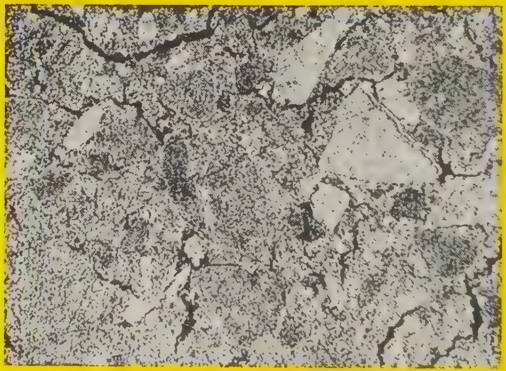
This unretouched photomicrograph shows the good uniformity and distribution of particles. This material is highly machinable, but as a result of its crystal structure, machined surfaces are relatively coarse.

GRADE SPK GRAPHITE →

Excellent uniformity of small particles and crystal structure, combined with *high density*, provides a *strong, highly machinable* graphite electrode.

GRADE L113SP CARBON →

Due to its lack of uniformity of particle sizes and crystal structure, and considering its brittleness and low strength, carbon is relatively difficult to machine and use without high breakage.



RESISTANCE TO OXIDATION

Grade SPK Graphite lends confidence to spectroscopic results. Because of its resistance to oxidation, it consistently provides minimum background effect.

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UNION CARBIDE

Spectral Line— *(Continued from page 9)*

Dr. Herbert Broida has been appointed a Senior Research Fellow at the National Bureau of Standards. Dr. Broida has received many awards and honors, including the Department of Commerce Gold Medal Award in 1960 for Exceptional Service, in which he was cited for "outstanding scientific and administrative leadership in the direction of the Bureau's Free Radicals Research Program". This referred to a 3-year program to increase the fundamental knowledge of the highly reactive molecular fragments designated as free radicals. Other honors included a year of study at Cambridge, 1959-60, on a National Science Foundation Senior Postdoctoral Fellowship; the Arthur S. Fleming Award in 1956 as one of five outstanding young scientists in government; and a year of study, 1952-3, at Imperial College of Science and Technology in London as a Guggenheim Fellow.

Dr. Broida, formerly chief of the free radicals research section at the Bureau, has conducted research in the fields of high temperature spectroscopy, combustion kinetics, medical instrumentation, as well as the trapping of free radicals.

Born in Aurora, Colo., in 1920 Dr. Broida has received three degrees in physics: B.A. in 1944, University of Colorado, M.A. and Ph.D. in 1946 and 1949, respectively, from Harvard University. He served as a teaching fellow and research assistant at Harvard while doing graduate work at that institution, and earlier, had taught physics at Wesleyan University.

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Dr. John K. Taylor, research chemist, whose entire 32-year professional career has been with the National Bureau of Standards, has been appointed Chief of the Applied Analytical Research Section of the Bureau's Analytical and Inorganic Chemistry Division.

The Applied Analytical Research Section is concerned with the development and application of methods for the analysis and characterization of solids, liquids, and gases. Both classical and instrumental methods of analysis are employed, using both macro-and micro-techniques. The Section also provides analytical consultation services and assistance for other Bureau programs. A major part of Dr. Taylor's work at the National Bureau of Standards has been concerned with the application of physical methods to chemical analysis, including the polarographic method and the limitations of voltammetric processes. In 1960, he was awarded the Department of Commerce Silver Medal in "recognition of his contributions to accurate electro-chemical methods of analysis".

Born in Mt. Rainier, Md. in 1912, Dr. Taylor received his B.S. in chemistry from George Washington University in 1934. He received his M.S. and Ph.D. from the University of Maryland in 1938 and 1941, respectively. Dr. Taylor joined the Bureau in 1929 as a laboratory aide. Later, after a 7-year period in the Optical Instruments Section, he transferred to the Chemistry Division as a physical chemist, where he has since remained.

Throughout his professional life, Dr. Taylor has been an active supporter of science education. He is a member and past chairman of the Joint Board on Science Education of the Washington Academy of Sciences and is editor of "The Reporter", a newsletter for science and mathematics teachers. He is a member of the Board of Directors of the Metallurgy and Ceramics Foundation, and is also a part-time member of the Chemistry faculty of American University.

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John L. Hague has been appointed Chief of the Standard Reference Materials Section at the National Bureau of Standards. The Standard Reference Materials Section will play an important role in the Bureau's Standard Materials program. In this program, the Bureau makes available for distribution to science and industry more than 550 different standards of metals, ores, ceramics, chemicals, hydrocarbons, and more recently added radioactive materials, uranium isotopes, lithium ores, aluminum refractories, and phosphor materials. Standard materials are used to check, evaluate, and standardize methods of analysis and analytical techniques and substances, and to develop new and improved methods and instruments.

Mr. Hague joined the Bureau staff in 1930 as a laboratory apprentice. In 1932 he transferred to the Norfolk Naval Shipyard and three years later was in charge of the Foundry Control Laboratory for ferrous and non-ferrous alloys. He returned to the National Bureau of Standards in 1935 and in 1951 became Assistant Chief of the Analytical Chemistry Section.

Born in Kansas City, Mo., in 1912, Mr. Hague received his B.S. degree in chemistry in 1938 from George Washington University and has done graduate work there. In 1958, Mr. Hague received the Department of Commerce Meritorious Service Award "for outstanding contributions to methods of analysis necessary for controlling the production of complex metal alloys".

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James F. Murphy has been appointed Supervisor of Spectrochemistry at the Inland Steel Company in East Chicago, Indiana. Mr. Murphy was formerly Chief Chemist at ACF Industries in Albuquerque, New Mexico, where he was responsible for supervision of the Chemistry and Chemical Engineering sections of the Development and Test Engineering Department.

Mr. Murphy has been quite active in the American Society for Testing Materials. He is a member of Committees E-2, E-3, and E-13, and is chairman of Subcommittee VI of Committee E-2. He has had four suggested methods for spectrochemical analysis of various materials published in the 1960 edition of *Methods for Emission Spectrochemical Analysis* (ASTM).

Mr. Murphy attended Purdue University and received his B. S. in Chemistry from Ball State in 1948.

——— λ ———

Lawrence R. Pittwell is now Assistant Professor of Chemistry at the University of Addis Ababa, Addis Ababa, Ethiopia. He was formerly Chief Chemist at the Dominion Magnesium Ltd., Haley, Ontario, Canada. He also served as National Secretary-Treasurer of the Canadian Association for Applied Spectroscopy.

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Drs. Harry E. Weaver and *James T. Arnold* have been appointed to research posts in the Varian Associates' Instrument Division. Dr. Weaver has been named manager of optical pumping and cryogenics research, and Dr. Arnold has been appointed manager of research in geophysics and space physics.

Dr. Weaver is a native of Philadelphia. He attended Case Institute of Technology, Cleveland, Ohio; and Stanford University, where he received his Ph.D. in physics in 1952. Prior to joining Varian Associates in 1954, he served in research and teaching posts at Case Institute, Stanford and the University of Zurich, Switzerland.

(Continued on next page)

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Spectral Line—Cont'd

Credited with several patent disclosures in the field of electron spin resonance, Dr. Weaver has been active at Varian in research involving the use of nuclear magnetic resonance and electron paramagnetic resonance principles in instrumentation for spectroscopy. Until recently, he was employed at Varian's research laboratory in Zurich, Switzerland.

Dr. Arnold was born in China, where he lived for 15 years. He attended Oberlin College in Ohio and Stanford University, where he recevide his Ph.D. in physics in 1954. He served as a research associate to Dr. Bloch, spent a year doing research at CERN in Geneva, Switzerland, and was assistant professor of physics at Oregon State College when he joined Varian in 1958. During World War II, Dr. Arnold served two years as a test pilot for the U.S. Navy.

In his new post, Dr. Arnold will direct research leading to instruments for use in geophysics and space physics studies. Several Varian-developed instruments have already been rocketed into space to measure magnetic fields.

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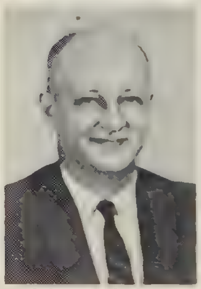

Carl A. Odening has been named administrative assistant, office of the president, by National Carbon Company, Division of Union Carbide Corporation.

Mr. Odening was graduated from the University of Pennsylvania in 1937 with a B. S. degree in electrical engineering, and joined National Carbon Company in the general engineering department in Cleveland. In 1939 he transferred to the research laboratories, and in 1940 he was assigned to the development laboratories in Niagara Falls.

From 1942 until 1953, Mr. Odening served in the production organization of National Carbon. His assignments in all of the company's plants included the position of plant manager of the electrode plant at Columbia, Tenn. In 1953 he was granted a Sloan fellowship for a year of advanced management training at Massachusetts Institute.

From 1954 until 1956, Mr. Odening was assistant to the general works manager of National Carbon Company in Cleveland, and since 1956 has served as director of development for the company in New York.

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(Continued on next page)

Sigurd Varian, 60, a founder of Varian Associates and an electronics pioneer, was killed October 11th when his light airplane crashed in a heavy sea off the west coast of Mexico.

A pioneer Pan American Airways pilot in Mexico, Sigurd as early as 1935 recognized the need for some method of preventing airplane crashes in fog, darkness or bad weather. When his brother Russell, then a graduate student in physics at Stanford University, conceived the idea of using short wave radio beams for this purpose, Sigurd left his job as a Pan American flight captain to throw his life's savings and full energies into a project to develop a practical device that would prove Russell's theory.

Granted the use of research facilities at Stanford, and assisted by Dr. David Webster and the late Dr. William W. Hansen, the Varian brothers spent three years on the project. The result was the klystron, a microwave device that made possible World War II radar. Since that time, radar has become an indispensable part of modern aviation.

In 1950, Sigurd and Russell Varian were awarded the John Price Wetherill Medal of the Franklin Institute of Philadelphia "in recognition of their foresight in anticipating the need for a converter of electromagnetic energy in a hitherto unexplored high frequency region of the spectrum, their energy and technical insight in developing for this purpose a practical device, the klystron, which has opened up new fields in communications applications and research."

Holder of numerous patents for inventions in microwave and other fields, Sigurd Varian was active in engineering and development work at Varian Associates until a few years ago. He retired to Puerto Vallarta, returning frequently to California for business reasons and to visit friends and family. Despite his retirement, Sigurd retained his keen interest in trying out new ideas. He continued inventing new devices of all kinds—from a precision high speed drill to a system of pumps, filters and heaters for his swimming pool.

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Dr. Samuel Jacobsohn passed away on Thursday, October 5, at the age of 63. Dr. Jacobsohn was President and Chief Physicist of The Gaertner Scientific Corporation, Chicago, Ill., and had been with this company over 40 years. He will be long remembered by his co-workers, customers and all with whom he came in contact for his wealth of scientific knowledge, considerate nature and his sincere appreciation of the arts.

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William C. McCosh has been named director of marketing for National Carbon Company, Division of Union Carbide Corporation. He succeeds Curry E. Ford who was recently appointed director of development for the company.

Mr. McCosh was graduated from the University of North Dakota in 1939, and joined National Carbon that year. Following several assignments in industrial sales, he was named manager of the central division of carbon products sales, with headquarters in Chicago, in 1954. In 1956 he was made manager of railroad and industrial brush sales in New York, and since 1960 he has served as manager of carbon products in the marketing organization.

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Curry E. Ford has been named director of development for National Carbon Company. He succeeds Carl A. Odening, who has been appointed administrative assistant in the office of the president.

Mr. Ford was graduated from Purdue University in 1933 with a B. S. degree in engineering, and received his M. S. degree from Harvard in 1934. He joined National Carbon in 1937, and was active in the development of carbon and graphite chemical process equipment.

In 1953 he was named manager of chemical products sales. He was appointed new products marketing manager in 1957, and since February, 1960, has been director of marketing.

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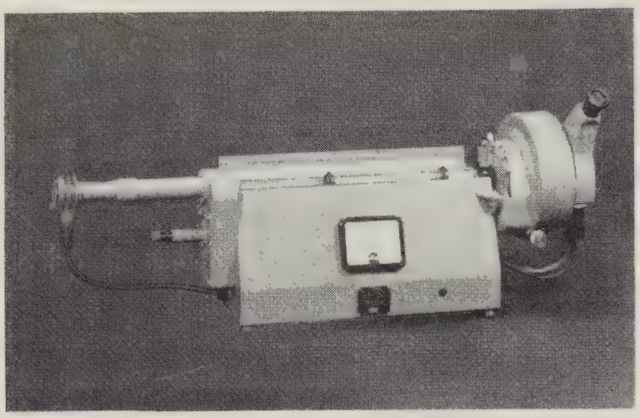

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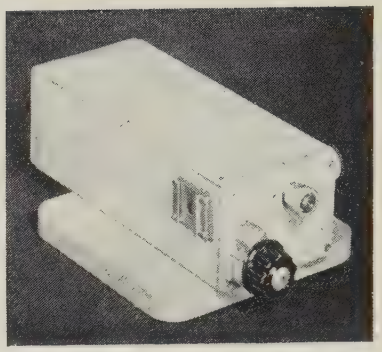

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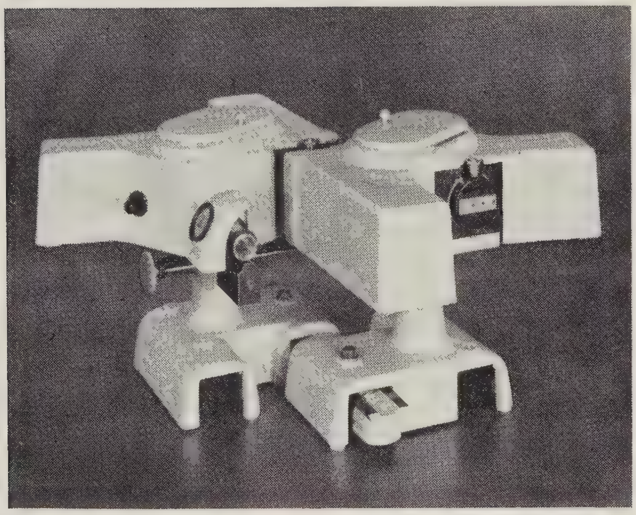
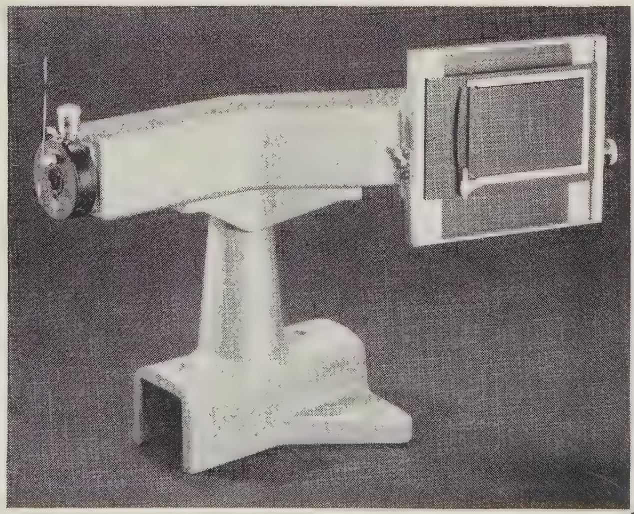


D 284—DOUBLE MONOCHROMATOR



E 584—SMALL PRISM SPECTROGRAPH

COMPACT GRATING MONOCHROMATOR

D 290—This new, low cost instrument utilizes a 15,000 l.p.i. plane grating covering a spectral range of 2000A to 10,000A with a dispersion of 70A/mm. Can be used vertically or horizontally. Its small size permits combining into other optical systems easily.

MICROPTIC PHOTOELECTRIC POLARIMETER

M 511—For research, requiring higher precision; for industrial laboratories where time, and operator fatigue, are important, a precision of .002° is obtained with an overall accuracy for any rotation of .005°. For use where the optical activity of the sample is to be measured. Range 0—360°, $-150°$ to $+150°$ sugar.

LARGE APERTURE DOUBLE MONOCHROMATOR

D 284—Rapid changing of 60° prisms permits wavelength coverage from 0.20 to 26.0μ. Used as an infrared spectrometer with a .01 second response Schwarz Thermopile, this instrument covers a region from .25μ to above 26.0μ. It may be separated for use as two single monochromators. These features, with the focal length of 330 mm. and an F/5 aperture, make this an exceedingly fine optical instrument.

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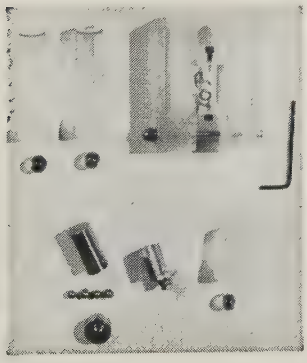
ACCESSORIES FOR NO. 6 include Plastic Balls & Vials (60 ml) to eliminate metallic contamination.

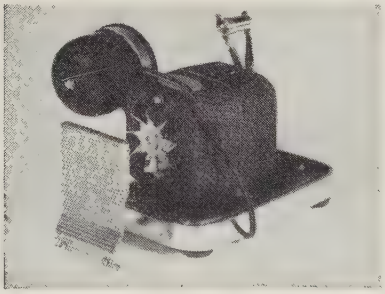
Stainless Steel Vials 6 ml or 10 ml and Rod Pestles or Ball Pestles. Tungsten Carbide 6 ml and 25 ml Vials and Rod or Ball Pestle.



ACCESSORIES FOR 5A OR 3A WIG-L-BUGS

3113 Adapter, for ½" dia. x 1" long vials.
3111 Vial, ½" x 1" polystyrene with cap.
3112 Ball-pestle, clear plexiglas, ⅜" dia.
3115 Adapter for ½" dia. x 2" long vials.
3116 Vial, ½" x 2" polystyrene with cap.
3113 Adapter for ½" dia. x 1" long vials.
3114 Vial, stainless steel ½" x 1" with ball-pestle ¼" dia.
3117 Vial, tool steel (hardened) ½" x 1" with steel ball-pestle ¼" dia.
3118 Vial, agate 9/16" dia. x 1 5/16" long 2 ml capacity with agate ball-pestle ¼" dia. (Requires 3115 adapter).

5A—Wonder Electric Mortar—the action is reciprocating in the form of a figure 8 swung through a 6 ½° arc at 3200 RPM, the ball pestle then strikes the end of the vial some 2000 times in ten seconds. Can be run continuously for 5 minutes (and this depends on the load) with a rest period between the 5 minutes to cool the motor completely.



3A—Model with Fan and 1 hour timer—same type of motor and action as 5A Model but this 3A can be run for 20-30 minutes with cooling off periods between to cool the motor.

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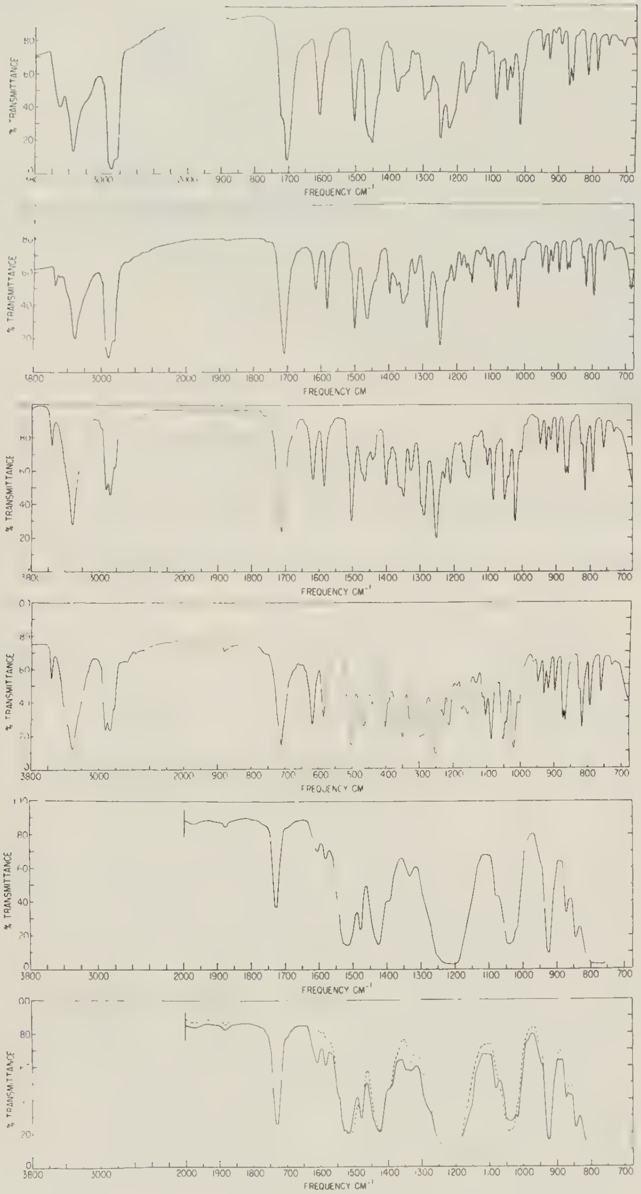

FIG. 1. 3,11β-DIHYDROXY-1,3,5(10)-ESTRATRIEN-17-ONE
A—Mineral oil mull spectrum: Polymorph I (top), B—Mineral oil mull spectrum: Polymorph II, C—Resolidified melt spectrum: Polymorph I, D—Resolidified melt spectrum: Polymorph II, E—Chloroform solution spectrum: Polymorph I, F—Chloroform solution spectrum: Polymorph II (---Chloroform blank)

limitations, since the compound solubility is not a limiting factor and resolidified melt spectra have essentially as many absorption bands as mineral oil mull spectra, with no "blanked-out" regions.

Figure 1 shows a comparison of the mineral oil mull, chloroform solution, and resolidified melt spectra of typical polymorphs of 3,11β-dihydroxy-1,3,5(10)-estratrien-17-one. The mull spectra are not identical but do show absorptions indicating that the same functional groups are involved. With this suspicion of polymorphism, the resolidified melt spectra are run, and the identity of these spectra establishes the fact that they are polymorphs. The chloroform solution spectra demonstrate the serious problem of sample insolubility, since only the 2 to 3 bands in the 1750 to 1600 cm^{-1} region of the spectrum allow any comparison.

The essence of the melt procedure is that different polymorphic forms of the same compound, treated under identical conditions of melting and cooling, will revert to that single polymorph favored by energy considerations. This approach requires complete melting, identical cooling rates, and the absence of foreign material that could provide nucleation sites favoring a particular polymorph. In practice, no difficulty from these latter factors has been observed. In compounds studied in the authors' laboratory, the lack of identity of spectra on resolidified melts has always proved to correlate with isomers or different structures.

The melt method obviously cannot be used in cases involving sample decomposition or rearrangement prior to or at the melting point of the compound. Rosenkrantz and Zablow (*8*) report that steroids melting below about 225°C are structurally unaltered during the preparation of melted films. This study has been extended to nonsteroids with a total of 35 compounds (both steroid and nonsteroid) being studied without difficulties due to decomposition.

Identification of Solvates

In many cases identification spectra can readily be obtained upon gases under a partial pressure of a few mm Hg (*14*). Many solvated compounds contain a 1:1 mole ratio of solvent to compound, which in the case of common solvents such as chloroform, acetone, and methylene chloride, means that a 200-mg sample will yield ample solvent in the gas phase for identification. Such positive characteristic spectral identification is a distinct advantage over the gas chromatographic method reported by Childs and Henner (*15*).

The low vapor pressure of water at room temperature necessitates a different approach for hydrates. In this case, the water can be condensed directly upon the salt plate and identified by its infrared spectrum as a liquid.

Gas spectra obtained from methylene chloride, methanol, ethanol, and acetone solvates have been identical to

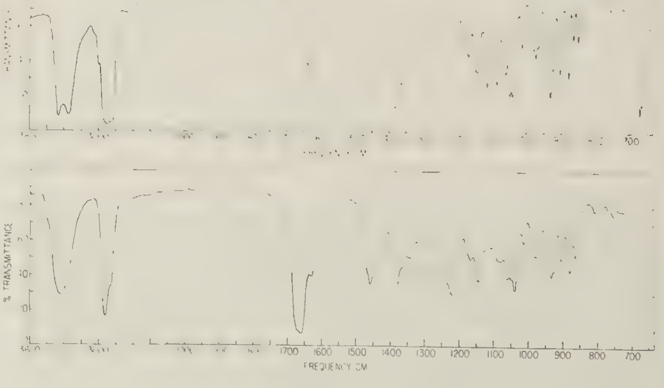

FIG. 2. 9α-FLUORO-11β,17β-DIHYDROXY-2α,17-DIMETHYL-4-ANDROSTEN-3-ONE

A—Benzene from solvated sample (top), B—Mineral oil mull spectrum of solvated sample, C—Resolidified melt spectrum of sample the reference spectrum of the corresponding solvent. Comparison of the spectra in Figure 2 of solvated 9α-fluoro-11β, 17β-dihydroxy-2α, 17-dimethyl-4-androsten-3-one, obtained on mull, melt, and solvent samples emphasizes the two positive aids for solvate identification offered by such a comparative procedure: 1) the solvent spectrum run in the gas phase, and 2) the removal of solvent bands from the spectrum of the resolidified melt.

Acknowledgment

The authors thank Dr. Robert W. Rinehart for his interest and most helpful discussions during the course of this work and in the preparation of the manuscript. The contribution of Dr. Stanley K. Derby in the early exploratory work is also acknowledged.

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Submitted December 22, 1960

Report of Nonmetallic Standards Committee Canadian Association for Applied Spectroscopy

Abstract

Two samples, a syenite rock and sulphide ore, are now available from the Nonmetallic Standards Committee of the Canadian Association for Applied Spectroscopy. Analyses are given along with tentative recommended values.

Purpose

The purpose of the Nonmetallic Standards Committee of the Canadian Association for Applied Spectroscopy is to evaluate the need for nonmetallic standards, particularly for the use of spectroscopists, and to foster the production of such standards if the need is justified. Accordingly, this committee initiated a test program to ascertain the demand for nonmetallic standards by issuing two nonmetallic standards (1) a sulphide ore and (2) a syenite rock sample.

This paper reports analyses which have been received to the time of writing. The samples are now available for sale and the demand for them will provide an indication of the need for such standards and will, perhaps, provide justification for a more permanent program of producing, certifying and issuing nonmetallic standards.

History

In January 1957 a preliminary questionnaire was sent to some 80 laboratories in Canada and 80 laboratories in the United States by W. H. Champ of the Geological Survey of Canada and W. H. Dennen of Massachusetts Institute of Technology. One of the purposes of this survey was to test the feasibility of the production of new nonmetallic standard samples to extend the coverage of the few now available. About 60 replies to the questionnaire were received, nearly all of which wanted some action to be taken to produce standard samples of this type. In order to foster the production of new nonmetallic standard samples, the Nonmetallic Standards Committee of the Canadian Association for Applied Spectroscopy was formed in September 1957 at the Fourth Ottawa Symposium on Applied Spectroscopy. Members of the committee are: C. L. Lewis, Chairman, Falconbridge Nickel Mines Limited, Richvale, Ontario; J. E. Burgener, Technical Service Laboratories, Toronto, Ontario; W. H. Champ, Geological Survey of Canada, Ottawa, Ontario; W. O. Taylor, Ontario Department of Mines, Toronto, Ontario; and G. R. Webber, Secretary, McGill University, Montreal, Quebec.

It was decided to carry out a test program of limited extent to ascertain the actual demand for nonmetallic standards with the hope that a demonstration of interest would make possible the establishment of a more extensive program for issuance of standards. Financial support was provided by the CAAS to permit the carrying out of this proposal. Two samples were obtained to serve as standard material, 500 lbs of sulphide ore from Falconbridge Nickel Mines Limited and 180 lbs of syenite rock from the Ontario Department of Mines. Analyses were solicited from a limited number of analysts. Sufficient results have now been obtained to justify a more widespread distribution of these samples. D. M. Shaw of the Department of Geology of Hamilton College, McMaster University kindly assisted the committee in its review of the analytical results. Revised lists of analyses will be issued when further results are obtained from other analysts. Up to the present time these samples have been sent to 28 analysts who have offered to contribute analyses.

History and Description of Samples

Sulphide Ore-1

The sample was collected at the Chemical Laboratory of Falconbridge Nickel Mines Limited, Falconbridge, Ontario. Sample rejects were saved during the Spring of 1958 until 500 lbs were accumulated in two small drums. These were sent to Falconbridge Metallurgical Laboratories at Richvale, Ontario. A grab sample of the material gave the following screen test: — 100 mesh: 93.34%, + 100 — 65 mesh: 5.36%, and + 65 mesh: 1.30%.

To ensure homogeneity, the sample was poured in two equal piles on a new sheet of plywood and each pile was riffled through a sample splitter to obtain first four 125 lb portions and then eight 62.5 lb portions. One such portion from each original pile was taken to make 125 lbs representative of the original 500 lbs.

TABLE I. SUMMARY OF ANALYSES FOR SULPHIDE ORE-1[a]

Element	*No. of Analyses*	*Analyses*	*Mean*	*Tentative Recommended Value*
		Rock Analysis Constituents %		
SiO_2	4	34.24,34.67,34.7,34.9	34.63	34.6
Al_2O_3	5	7.88,8.12,9.26,9.65,10.04	8.99	
Fe	4	23.5,23.62,23.7,25.18	24.00	23.6
MgO	5	3.71,3.94,4.10,4.14,4.49	4.08	
MnO	11	0.04,0.06,0.09,0.10,0.10,0.12,0.12, 0.13,0.13,0.14,0.18	0.11	0.11
CaO	5	3.67,3.74,3.87,3.96,4.16	3.88	3.9
Na_2O	4	0.85,0.86,0.90,1.01	0.91	0.9
K_2O	3	0.55,0.62,0.70	0.62	
H_2O-	2	0.05,0.09	0.07	
H_2O+	2	2.77,3.09	2.93	
TiO_2	10	0.50,0.65,0.69,0.74,0.75,0.77,0.83, 0.83,0.89,1.0	0.77	0.77
P_2O_5	2	0.09,0.11	0.10	
S	4	11.9,12.06,12.17,12.2	12.08	12.1
		Minor Elements, ppm		
Ag	9	2.5,3.3,4.4.2,4.9,5,5,6	4.2	4
As	2	400,560	480	
Au	1	0.09	0.09	
B	1	20	20	
Ba	6	97,200,200,260,290,310	226	
Bi	1	8	8	
Co	9	300,300,500,500,500,590,650, 680,680	522	
Cr	5	230,290,300,530,1000	470	
Cu	8	4000,7000,8200,8200,8400, 8700,8840,12000	8168	8500
Ga	2	13,15	14	
Li	1	10	10	
Mn	11	300,500,700,774,774,900, 929,980,1000,1100,1400	851	
Mo	1	10	10	
Ni	7	6000,7000,13500,14000,15060, 15200,15200	12280	
Pb	9	200,210,220,250,280,280, 300,300,310	261	260
Pd	1	0.14	0.14	
Pt	1	0.24	0.24	
Rb	1	200	200	
Rh	1	0.09	0.09	
Sc	3	24,25,27	25	25
Sn	1	3	3	
Sr	3	100,130,170	133	
Ti	10	3000,3900,4100,4400,4500,4600, 5000,5000,5300,6000	4580	
V	6	110,150,210,220,220,262	195	
Y	2	20,23	22	
Yb	1	6.2	6.2	
Zn	5	160,200,210,350,550	294	
Zr	5	90,94,100,110,151	109	109
U_3O_8	1	2	2	

[a] Values reported as less than a specified value or as ranges have not been included in the above tabulation.

The 125 lb sample was placed in a small drum and turned for 24 hr on rolls at a speed calculated to give cascade action. It was then returned to the original plywood sheet and riffled to obtain 400 portions of about ¼ lb each. These were placed in 4 oz, wide mouth, glass jars covered with bakelite screw caps.

The sample, transportation, and mixing were donated by Falconbridge Nickel Mines Limited. The final splitting and bottling were done by members of the Toronto-Hamilton section of the CAAS.

Homogeneity tests were made by W. O. Taylor, Ontario Department of Mines, W. H. Champ, Geological Survey of Canada, and the staff of Falconbridge Metallurgical Laboratories.

A semiquantitative analysis was made by W. H. Champ and a qualitative analysis was made in the Falconbridge laboratory.

TABLE II. SUMMARY OF ANALYSES FOR SYENITE ROCK-1[a]

Element	*No. of Analyses*	*Analyses*	*Mean*	*Tentative Recommended Value*
		Rock Analysis Constituents %		
SiO_2	3	58.71,59.5,59.65	59.29	
Al_2O_3	3	8.53,8.93,10.5	9.32	
Fe_2O_3	3	1.85,2.22,2.46	2.18	
FeO	3	5.31,5.80,7.12	6.08	
Fe	1	5.94	5.94	
MgO	3	3.25,4.01,4.81	4.02	
MnO	8	0.35,0.36,0.39,0.41,0.44,0.45, 0.46,0.50	0.42	0.42
CaO	3	10.01,10.1,10.62	10.24	
Na_2O	3	2.74,2.94,3.29	2.99	
K_2O	3	2.39,2.80,2.94	2.71	
H_2O-	2	0.18,0.23	0.21	
H_2O+	2	0.42,0.50	0.46	
TiO_2	8	0.37,0.42,0.44,0.47,0.48,0.52, 0.62,0.83	0.52	
P_2O_5	3	0.16,0.18,0.23	0.19	
U_3O_8	4	0.22,0.285,0.30,0.31	0.28	
S	1	0.07	0.07	
CO_2	2	0.32,1.33	0.83	
		Minor Elements, ppm		
Ag	3	0.5,1.4,1.9	1.3	
B	6	58,64,80,100,120,320	124	
Ba	6	130,230,240,300,330,380	268	
Be	6	14,16,19,20,26,34	22	22
Ce	1	1100	1100	
Co	6	15,17,18,20,20,23	19	19
Cr	6	40,51,60,70,72,73	61	61
Cu	7	13,15,19,20,21,37,40	24	
Ga	5	10,10,20,20,21	16	
La	4	200,230,320,400	288	
Li	2	100,115	108	
Mn	8	2700,2800,3000,3200,3400, 3500,3600,3900	3260	3260
Nb	3	70,120,240	143	
Ni	6	20,30,37,50,59,82	46	
Pb	6	400,450,500,510,550,870	547	480
Rb	3	170,290,300	253	
Sc	5	12,15,15,19,20	16	16
Sn	2	6,20	13	
Sr	5	190,260,410,500,750	422	
Th	3	610,1400,1800	1270	
Ti	8	2200,2500,2640,2800,2900,3100, 3700,5000	3100	
U	4	1900,2400,2500,2600	2350	2400
V	6	50,55,69,96,100,104	79	
Y	5	300,410,460,660,700	506	
Yb	4	63,70,73,110	79	
Zn	3	70,180,350	200	
Zr	6	1500,2500,2900,2900,3300,3900	2833	3100

[a] Values reported as less than a specified value or as ranges have not been included in the above tabulation.

Syenite Rock-1

Approximately 180 lbs of syenite rock collected from four different properties in the Bancroft Area, Ontario were donated by S. E. Wolfe, Mining Engineering Department, University of Toronto. This material was crushed at the University of Toronto and was pulverized at the Provincial Assayers' Laboratory, Ontario Department of Mines, in a Braun pulverizer with the plates set to yield at least —100 mesh. The sample was not screened.

The sample was placed in a small drum and was turned for 24 hr on rolls at a speed calculated to give cascade action. It was then placed on a new sheet of plywood and was split in a riffle to obtain 400 portions of about ¼ lb each. These were bottled in 4 oz, wide mouth, jars and covered with bakelite screw caps.

The material was provided by the Ontario Department of Mines and the work of splitting and bottling was contributed by members of the Toronto-Hamilton section of the CAAS.

Homogeneity tests were made by W. O. Taylor, Ontario Department of Mines, and W. H. Champ, Geological Survey of Canada and Mr. Champ also made a semiquantitative analysis of the material.

Analytical Results

Analytical results are reported in Tables I and II. The committee plans to issue revised results periodically as more analyses are received.

List of Participating Laboratories

The laboratories cooperating include: Control Analysis Section of the Extraction Metallurgy Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Ontario (J. C. Ingles); Department of Geological Sciences, California Institute of Technology, Pasadena, California (A. A. Chodos and Elizabeth Godijn); Department of Geology, Hamilton College, McMaster University, Hamilton, Ontario (D. M. Shaw and J. N. Weber); Department of Mineral Engineering, Stanford University, Stanford, California (H. J. Moore II, C. M. Taylor, and Kurt Servos); Falconbridge Nickel Mines Limited, Richvale, Ontario (C. L. Lewis, L. L. James, and N. M. Sine) and Falconbridge, Ontario (M. H. Cornett); Indiana Geological Survey, Bloomington, Indiana (M. E. Coller and R. K. Leininger); Miller Research Laboratories, Queen's University, Kingston, Ontario (J. E. Hawley, L. G. Berry, J. G. MacDonald, and Frank Dunphy); Ontario Department of Mines, Toronto, Ontario (R. Smith, I. Aloe, J. Stevenson, B. Eckenbach, and W. O. Taylor); U. S. Geological Survey, Denver, Colorado (F. C. Canney, U. Oda, and H. L. Neiman) and Branch of Analytical Laboratories (A. T. Myers and N. M. Conklin).

Availability of Samples

Both the sulfide ore and syenite rock samples may be ordered at a cost of $5 per bottle from:

G. R. Webber
Department of Geological Sciences
McGill University
Montreal, Quebec, Canada

Funds received from the sale of these samples will be used to reimburse the CAAS for its support of this analytical program and to foster further work of this nature.

It would be appreciated if results of any analyses made on these samples could be sent to the above address in order that they may be incorporated in future revised analytical lists.

Submitted May 19, 1961

Application of the Tape Technique to the Spectrographic Determination of Mg, Mn, P, and B in Unashed Plant Material

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Abstract

A multisource unit and tape machine were employed in the determination of Mg, Mn, P, and B in unashed plant material. The use of working curves from synthetic standards was investigated and found suitable in the case of P. Analysed natural standards were used for obtaining working curves for Mg, Mn, and B. In all cases carbon powder was used as buffer material. The coefficients of variation determined for Mg, Mn, P, and B were 1.44, 4.23, 1.04, and 2.57% respectively.

Introduction

A very important drawback in Agriculture Science is the lack of a fast and reliable method for the determination of macro- and micro-elements in plant material. In the conventional spectrographic analysis of ashed plant material the preparation of the final form of the sample is time-consuming and tedious, and it presents more opportunity for human errors.

The analysis of unashed plant material by modern spectrographic methods has received very little attention up to the present. Munst and Melstedt (*1*) developed a method for the determination of macro- and micro-elements in unashed plant material, employing a high voltage spark and briquetted samples. However, their method was very unsatisfactory, since the accuracy was only in the order of ±20%. With the invention of the tape machine by Danielsson (*2*) the spectrographic analysis of unashed plant material again presented itself as a possibility.

In the tape method the sample in powder form is continuously fed onto an adhesive tape, which is passed through a spark gap at such a rate that every single spark always strikes new material. By standardizing the conditions under which the spark occurs and employing a satisfactory technique for uniform sample preparation, the reproducibility obtainable is quite exceptional.

It was thus decided to reinvestigate the spectrographic analysis of plant material in the unashed form by using the tape method of introducing the sample into the source.

Experimental

The tape machine used in this project was built in our workshop according to the design of Danielsson, Sundkvist, and Lundgren (*2*). It differs somewhat in detail from the machine now commercially available.

A photograph of a front view of the tape machine is shown in Figure 1. Rolls of folded tape are loaded on spool, *1*. The tape passes over the centering wheels *2* and *3*, underneath the sample feeding device, *4*, and over guide

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*Department of Physics

†Stellenbosch-Elsenburg College of Agriculture. This research was carried out in the Physics Department as partial fulfillment of the requirements for the M.Sc. degree in Physics.

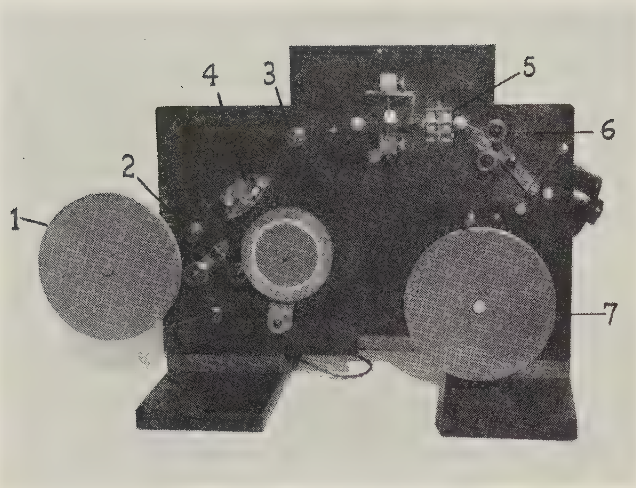

FIG. 1. THE MODIFIED TAPE MACHINE

pins through the spark gap. The tape now passes through an "extinguishing" device, *5*, and is clamped between the pulling wheels, *6*. The used tape is taken up by the discharge spool, *7*. The outer pulling wheels are made of brass and the inner one of gear-bakelite. This inner wheel is pressed against a rim in the outer wheels by means of a spring in such a way that the pull is exerted only on the folded edges while the central adhesive part passes through without any obstruction. All wheels and other devices were mounted on a sheet of Delaron** board in this experimental design.

The tape was folded to make it mechanically stronger, to reduce sample consumption and to obtain a more definite location of the spark. The spark discharge occurs where the gap resistance is the lowest, i.e. through the central portion of the tape, thereby increasing the reproducibility of the exposures. Two types of tapes were used, namely commercial Sellotape‡, 1.27 cm wide, and special A.R.L.†† tape. Both were folded to the same final width of 0.30 cm. It was later decided to use the A.R.L. tape exclusively because of its higher purity especially in magnesium. In initial experiments the commercial rolls of Sellotape were used directly. It was found however that the tape was not evenly packed on the roll, and that the rolls were deformed with the result that different amounts of strain were exerted irregularly on the tape while being pulled through the spark gap. This also resulted in changes in the speed of the tape so that some sparks occurred at shorter distances on the tape, disrupting the ideal spectrographic conditions and burning a hole through the tape.

These problems were solved by folding the tape before usage. This was done by mounting the stock rolls on a special spool, *8*, as shown in Figure 2. The tape passes over the folding wheel, *9*, and is diverted past the charging device over wheel *10* to the pulling wheels. The tape is thus folded in this process, but the central adhesive portion is kept perfectly free from any sample. The tape is packed into very neat, uniform rolls on the spool, *7*, which is driven by means of a spring belt from the same motor, *M*, which operates the pulling wheels. The folded rolls are rotated through 180° and inserted into the feeding spool, *1*, as previously described. This spool is provided with a friction coupling to ensure uniform pull on the tape, while it is fed through the spark.

The sample container is made from acrylic sheet (Perspex) and fits into a larger acrylic sheet pocket. A vibrator mechanism operated from the 50-cycle main regulates the flow of sample on the tape. A suction device for the removal of excess sample is also incorporated into the charging unit.

The "extinguishing" device consists of two glass plates, about 2 cm x 5 cm, with a gap between them of approximately one mm, through which the tape passes. This device prevents the burning of the tape when high energies are used. The tape moves at a speed of 5.1 cm/sec.

The tape machine is not mechanically connected to the optical bench to avoid the introduction of any possible vibration to the spectrograph.

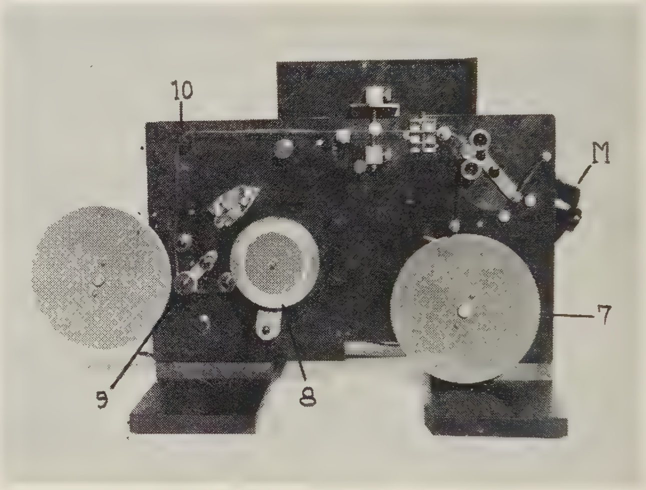

FIG. 2. THE TAPE MACHINE IN THE FOLDING POSITION

Spectrographic Conditions

The essential operational data are given in Table I. Preliminary exposures showed a relatively high background to be associated with the proposed tape method when the spark burned in air. To investigate this problem, the tape machine was adapted to permit the use of different atmospheres. A brass cylinder, 14 cm diam., fitted with O-rings on either side, was clamped against the Delaron framework. The tape passed through adjustable aluminum slits on the sides of this tube. The front cover carried a tube extension fitted with a quartz window. The inert gas was fed into the chamber from the far side through the Delaron board and a continuous flow of gas was maintained from the cylinders. A number of exposures were thus made in argon and nitrogen atmospheres to study the atmospheric gas effect. Unashed potato samples were used in all cases.

The intensity of the spectrum decreased considerably in the argon atmosphere. The lines B 2497, P 2553, and Mn 2576 were not visible, while the background still gave photometer readings of 90 and less in the densest step of the spectrograms. From this it was evident that an argon atmosphere was unsuitable for increasing line to background ratio in this case.

In the nitrogen atmosphere the lines B 2497, P 2553, and Mn 2576 were visible, although the intensities were

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**Delaron board, Fraser & Chalmers (S.A.) (Pty.) Ltd., P.O. Box 894, Cape Town, South Africa

‡"Sellotape" supplied by Adhesive Tapes S.A. (Pty) Ltd., P.O. Box 2596, Cape Town, South Africa

††A.R.L. Scotch Tape supplied by Minnesota Mining & Manufacturing Co. Ltd.

TABLE I. APPARATUS AND SPECTROGRAPHIC OPERATION CONDITIONS

Electrodes: Sample	¼ in. diam. graphite rod with tapered end centrally below A.R.L. Adhesive tape carrying unashed plant material
Counter	3/16 in. diam. graphite rod, with tapered end
Analytical gap, mm	5.0
Excitation unit	Self constructed Low Voltage Multisource Unit with 1000 V on storage condenser
Triggering	1 microsec pulse of 10,000 V peak, approx.
Form of discharge after triggering	Damped oscillations of approx. 2500 c/s, log decr. 0.7 approx.
Capacitance, μfd	6.0
Inductance, μh	750
Resistance, ohm	1.0
Discharge current, amp	80, Peak (approx.)
Repetition frequency	50/sec
Spectrograph	Hilger Medium Quartz Spectrograph
Wavelength range, A	2200 to visible
Slith width, μ	30
Filter	Hilger stepped neutral transmission filter transmission ratio 1:1.5
Optical system	Arc imaged on diaphragm of 6 mm aperture situated between slit and source. This diaphragm focussed on collimator lens.
Photographic emulsion	Ilford Ordinary N.30
Developer	Kodak D-19 B, 18°C, 5 min
Plate calibration	Variation of two-line method as described by Churchill (6) and Strasheim (7)

much lower than in air. The loss in intensity could not be compensated for by the decreased background intensity. Furthermore the tape became sticky which interfered with the pulling wheels and caused the tape to break consistently. Owing to the above-mentioned difficulties, this line of investigation was abandoned, and further exposures were made with the spark in air.

Spectral Lines Employed

Preliminary investigations showed that the lines Mg 2782.974, Mn 2576.107, P 2553.28, and B 2497.733 would be suitable for quantitative work from the intensity point of view.

Tin has thus far been detected only in exceedingly small quantities in plant material, and was employed as internal standard in plant analysis by Pienaar (5). The tin lines, 2706.510 and 2546.552, have relatively low excitation potentials and do not differ much in wavelength from the selected spectral lines of B, Mn, Mg, and P. For these reasons it was decided to use the line Sn 2706.510 as internal standard for Mg 2782.974, while Sn 2546.552 was employed with Mn 2576.107, P 2553.28, and B 2497.733. Investigations showed that graphite, mixed in a 1:1 ratio with the samples, was the most acceptable buffering substance for the unashed plant material. In subsequent experiments $CaCO_3$ and Li_2CO_3 were also tried but tended to suppress the emission of the selected spectral lines.

The suitability of the tin lines as internal standard spectral lines for the selected analysis lines of B, P, Mn, and Mg was investigated by making a number of exposures of the same plant material and by determining the relative line intensities and relative intensity ratios of the various line-pairs for the different exposures. This sample was prepared by grinding together equal amounts of sample and graphite, using graphite powder which contained 4500 ppm Sn. Background corrections were made for all the lines, including the internal standard lines. From the graphical representation given in Figure 3 it is evident that the lines Sn 2706 and Sn 2546 are suitable as internal standards for the analysis lines given above.

Preparation of Synthetic Standards—Choice of Matrix Material

The standards used for obtaining a working curve should approximate as closely as possible the chemical composition and physical characteristics of the unknown samples (3). Owing to the difficulties usually encountered in finding accurately analyzed natural plant material samples, synthetic base mixtures were first investigated.

Approximately 90% by weight of plant material consists of organic compounds, and the rest being inorganic in nature. The main organic constituents of plant material are cellulose and hemi-cellulose (4). According to present theory the cellulose molecule is a long chain formed from glucose (pyranose form) by the loss of H and OH at the 1 and 4 positions respectively. On prolonged treatment with sulfuric acid, dextrins and finally glucose (95% yield) may be produced. Hemicelluloses are similar to cellulose, but when they are hydrolyzed several different hexoses and also pentoses are produced. Furthermore starch occurs in distinct grains in plants. On prolonged hydrolysis of plant starch, maltose and finally glucose is produced. Cellulose is not available as a fine powder. All attempts to grind cotton wool (95-98% pure cellulose) to powder, were unsuccessful. Since sugar and starch are chemically closely related to cellulose, an attempt was made to use them as base for the preparation of the synthetic standards.

A number of exposures were made, using powdered sugar and graphite as sample. The sugar was found to be free from Mg, Mn, P, and B. However, two difficulties arose: a) The sugar caused the tape to burn, and b) Due to the hygroscopic characteristics of the sugar, the charging pocket clogged.

Icing sugar was subsequently tried, but no improvement could be claimed.

Further investigations were thus concentrated on starch as base material. One g of starch was ground with one g of graphite powder in an agate mortar for 5 min, and a number of exposures made. None of the difficulties en-

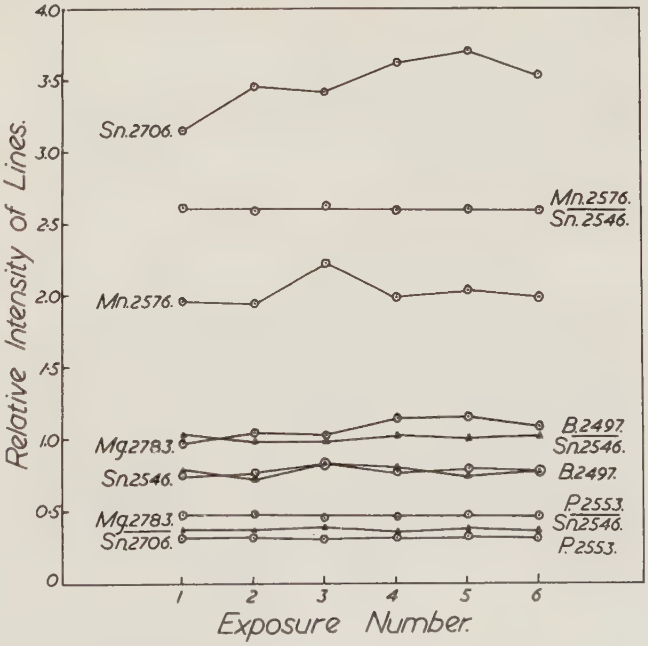

FIG. 3. RELATIVE INTENSITIES AND INTENSITY RATIOS AS A FUNCTION OF THE NUMBER OF EXPOSURES

countered with sugar as a base were experienced. The background intensity was approximately the same as when natural plant samples were used. Starches produced by four different manufacturers of chemical reagents were investigated for their purity with respect to Mg, P, and B. It was found that the AnalaR grade starch manufactured by Hopkins and Williams§ contained the least impurities.

Synthetic Standards

A closer approximation to natural plant material was effected by adding the major inorganic constituents of plant material in specpure powder form so that the composition of the base was 90% organic (starch) and 10% inorganic. This was done by adding 0.75 g K_2SO_4, 0.50 g K_2CO_3, 0.125 g NaCl, 1.625 g $CaCO_3$, and 1.00 g SiO_2 to 36 g of the selected starch. This mixture was ground in an agate ball mill for three hr to assure good mixing. A synthetic standard containing maximum concentrations of the various elements was prepared by mixing 2.30 mg Specpure H_3BO_3, 198.4 mg Specpure KH_2PO_4, 16.32 mg Specpure MgO, and 0.556 mg Specpure Mn_3O_4 with 1.7825 g synthetic starch base in an agate mortar for 5 min. This standard thus contained 200 ppm B, 2.26% P, 0.50% Mg, and 200 ppm Mn and was diluted with the synthetic starch base by factors of 2, 4, 8, 16, 32, and 64. This set of standards thus covered the following concentration ranges: Mg, 0.0078 - 0.5%; Mn, 3.125 - 200 ppm; P, 0.0354 - 2.26%; and B, 3.125 - 200 ppm.

Samples for sparking were prepared by grinding 0.2 g of a specific standard with 0.2 g graphite buffer mixture. These prepared samples were stored in glass vials until used.

The Working Curves

Duplicate exposures of the synthetic standards were made. New electrodes were used for every exposure. A single preliminary curve sufficed for all the lines used. Background corrections were made for the analytical elements and internal standard lines. The equation obtained for the regression line of the P working curve is as follows: P 2553/Sn 2546 : $Y_c = 0.720\ X + 0.0936$, Range: 0.07 to 3.0%, where Y_c is log (Rel. Int. ratio) and X is log (Concn.).

The working curves for Mg, P, and B showed a slight upward curvature at low concentrations. This is due to the residual magnesium, phosphorus, and boron in the tape and graphite. These impurities are constant for all analyses, and no corrections are needed for them, since they influence the working curves and actual analyses to the same degree.

Preparation of Plant Material Samples

The method of preparation of the plant material samples is analogous to the method employed by Pienaar (5).

The green leaves were washed in water containing Tepol¶ and rinsed with distilled water. The leaves were dried in an oven, supplied with an air current, at 70°C for a period of 24 hr. The dry leaves were ground in a porcelain ball mill for 10-20 min to a homogeneously fine powder and again dried at 70°C for 12 hr. The samples were then stored in airtight bottles. This same technique was used to prepare the pea samples.

The samples were prepared for sparking by grinding 0.2 g plant material with 0.2 g graphite containing the internal standard in an agate mortar for 5 min. This amount of sample was enough for duplicate exposures of 60 sec each.

Investigation of Working Curve Shift

A number of samples were analyzed to determine any shifts that might occur in the working curves owing to the use of synthetic standards.

Analyzed samples of prune, peach, apricot, and grape leaves were obtained from the Western Province Fruit Research Station. The Stellenbosch-Elsenburg College of Agriculture supplied analysed pea samples.

The peas were chemically analyzed for all four elements. The magnesium and manganese content of the other samples was determined spectrographically by the dc arc method developed by Pienaar (5), while the phosphorus and boron were determined chemically.

An appreciable shift of the working curve for magnesium was evident. On the average the values obtained with the tape method were 49.6% lower than the values obtained by the other methods. Similarly the manganese values obtained were about 258% higher than the chemical values. The phosphorus values were 3.6% too low, while the boron values were 18.3% too low.

In the plant the magnesium atom forms the centre of a huge molecule. The magnesium would thus be more difficult to excite than in MgO, which was added in the synthetic samples. This fact could account for the low values obtained.

The values obtained for boron are too low. This could be expected. Boron was added to the synthetic standards as Boric Acid. This is a relatively unstable compound. In plants however, boron is believed to be present in the form of borates which are chemically much more stable than boric acid. Phosphorus occurs as phosphates in plant material, but was also added as such, and this could account for the relatively good accuracy of the phosphorus determinations.

The manganese excitation is much stronger in plant material than in the synthetic standards. No explanation can be offered for this phenomenon.

Two conclusions concerning the synthetic standards were reached: a) The synthetic standards appear to be unsuitable for the preparation of working curves for Mg, Mn, and B if the recommended values of the analyzed samples are accepted as correct, b) Phosphorus determinations of sufficient accuracy are possible by using the synthetic standards.

It was consequently decided to use analyzed natural samples as standards for the preparation of working curves for Mg, Mn, and B.

Working Curves with Natural Standards

In order to obtain working curves for Mg, Mn, and B a number of analyzed samples were prepared for sparking as already described. It was necessary to calculate the regression lines because an appreciable scattering of the points was observed.

The equations obtained for the Mg, Mn, and B regression lines are as follows:

Mg 2783/Sn 2706;
$Y_c = 0.8287X - 0.2586$, Range: 0.08 to 1%
Mn 2576/Sn 2546;
$Y_c = 0.8313X - 1.4688$, Range: 5 to 1,000 ppm.
B 2497/Sn 2546;
$Y_c = 0.776X - 1.341$, Range: 10 to 100 ppm.

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§Hopkin & Williams Ltd., Chadwell Heath, Essex, England

¶The Shell Chemical Distributing Co. of S.A., Ltd., Johannesburg, South Africa

TABLE II. STANDARD ERRORS OF ESTIMATE AND CORRELATION COEFFICIENTS FOR MG, MN, AND B IN NATURAL STANDARDS AND FOR P IN SYNTHETIC STANDARDS

Line pair	r^a	$s_y{}^a$	$S_y{}^a$
Mg2783/Sn2706	0.965	0.2202	0.05776
Mn2576/Sn2546	0.985	0.4321	0.07457
B2497/Sn2546	0.982	0.1367	0.02583
P2553/Sn2546	0.999	0.3694	0.0085

[a] r = correlation, S_y = standard deviation of y-values, and S_y = standard error of estimate

As an estimate of the accuracy of the determinations possible when these and the phosphorus regression lines were used, the standard error of estimate, S_y, and coefficient of correlation, r, were calculated. The results are presented in Table II.

TABLE III. SUMMARY OF UNASHED PLANT MATERIAL SAMPLES ANALYZED WITH THE TAPE METHOD[a]

Peas[b]

Mg, %		*Mn, ppm*		*P, %*		*B, ppm*	
Chem.	*Tape*	*Chem.*	*Tape*	*Chem.*	*Tape*	*Chem.*	*Tape*
0.158	0.145	23	20	0.400	0.420	8.0	8.0
0.156	0.165	23	20	0.467	0.480	8.5	8.4
0.160	0.160	22	19	0.465	0.440	8.0	7.8
0.160	0.160	22	19	0.448	0.440	7.5	7.6

Apple leaves[c]

Mg, %		*Mn, ppm*		*P, %*		*B, ppm*	
Arc	*Tape*	*Arc*	*Tape*	*Chem.*	*Tape*	*Chem.*	*Tape*
0.28	0.295	93	105	0.190	0.195	34	37
0.34	0.350	39	53	0.181	0.170	34	33
0.25	0.335	87	100	0.150	0.130	78[a]	36

Apricot leaves[c]

Mg, %		*Mn, ppm*		*P, %*		*B, ppm*	
Arc	*Tape*	*Arc*	*Tape*	*Chem.*	*Tape*	*Chem.*	*Tape*
0.56	0.49	136	125	0.202	0.143	90[a]	23
0.78	0.85	159	210	0.174	0.165	47	46
0.82	0.90	162	144	0.159	0.090	73[a]	26

Grape leaves[c]

Mg, %		*Mn, ppm*		*P, %*		*B, ppm*	
Arc	*Tape*	*Arc*	*Tape*	*Chem.*	*Tape*	*Chem.*	*Tape*
0.64	0.51	211	180	0.166	0.130	58[a]	41
0.38	0.47	314	500	0.152	0.165	33	34
0.46	0.50	79	75	0.437	0.435	42	44
0.35	0.37	219	250	0.344	0.345	51	47
0.09	0.09	950	650	0.149	0.110	13	13

Pear leaves[c]

Mg, %		*Mn, ppm*		*P, %*		*B, ppm*	
Arc	*Tape*	*Arc*	*Tape*	*Chem.*	*Tape*	*Chem.*	*Tape*
0.62	0.52	44	44	0.176	0.150	27	29
0.15	0.12	11	10	0.365	0.340	38	35
0.19	0.18	5.0	5.2	0.262	0.230	37	34

[a] No correlation between the B values recommended by the W.P.F.R.S.[c] and those recommended by the S.E.C.A.[b] could be found.

[b] Analyzed at the Stellenbosch-Elsenburg College of Agriculture.

[c] Analyzed at the Western Province Fruit Research Station.

Applicability of the Method to the Analysis of Plant Material

The concentrations of the elements B, P, Mn, and Mg in different types of plant material samples, which were analyzed in the above-mentioned laboratories, were also determined by the tape method in order to ascertain the applicability of this method. The working curve prepared with synthetic standards was used in the determination of phosphorus, while the curves obtained with natural standards were used for magnesium, manganese and boron.

The results obtained in this investigation are listed in Table III. At least duplicate analyses were made of all samples. The values obtained with other methods are also listed for comparison.

TABLE IV. REPRODUCIBILITY OF THE MG, MN, P, AND B DETERMINATIONS

Element	*Coefficient of variation %*
Mg	1.44
Mn	4.23
P	1.04
B	2.57

Reproducibility

The reproducibility of the analyses was determined as follows: A sample of prune leaves was exposed as a monitor on every plate taken. This sample was freshly prepared for each plate. Finally a number of these monitor samples was prepared separately and exposed on the same plate. The coefficient of variation for the various elements determined are given in Table IV. These results confirm the much higher reproducibility of the tape method in comparison with for example, the dc arc method, and prove the suitability of this method for the analysis of unashed plant material.

Conclusion

From a routine point of view, this tape method has definite advantages. It calls for a minimum of sample preparation, which eliminates the time-consuming procedure of ashing and reduces the number of weighings. The reproducibility of the preparations is accordingly improved, since the sources of possible errors are limited.

The figures quoted in Table II-IV indicate conclusively that, provided accurate and reliable analyses of natural standards are available, the tape method described above can be used with advantage for the analysis of plant material.

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The Analysis of Uranium Alloys Using a Hollow Cathode*

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Abstract

This paper describes investigations that have been made with the Schüller-Gollnow water-cooled hollow-cathode tube for the purpose of using it as an analytical source to analyze fuel materials employed in nuclear reactors. A number of unirradiated normal and enriched uranium alloys have been tested with the above tube using a 21-ft, Wadsworth, Jarrell-Ash grating spectrograph and photographic plates. Results obtained for molybdenum, ruthenium, rhodium, and palladium, in the range of 2.5, 2.0, 0.3, and 0.2% respectively, compare favorably with the point-to-plane technique.

Introduction

The engineering of nuclear reactors has presented many analytical problems to the spectrographic laboratory. New uranium alloys are being fabricated and irradiated constantly. To expedite the loading of reactors, a quick and accurate method of analysis of irradiated and unirradiated fuel material is required.

Presently, the point-to-plane technique is being used satisfactorily at this laboratory for the analysis of normal and enriched unirradiated alloys (1). A metal sample having a flat machined surface approximately ⅛ in. diam. is sampled directly using a high voltage condensed ac spark and a ⅛ in. pointed graphite rod as a counter electrode. Comparison of spectrographic and chemical results on 24 alloys containing approximately 2.5% Mo, 2.0% Ru, 0.3% Rh, 0.2% Pd and 2.5% Zr shows a standard relative deviation of spectrographic from chemical results of ±3.9, ±4.5, ±7.2, ±6.6, and ±7.5% respectively. To meet the challenge of analyzing these alloys spectrographically on a routine basis after they have been irradiated in a reactor, new techniques and equipment were investigated, since it is expected the irradiated alloys will be too radioactive to handle using the present point-to-plane technique.

The use of a hollow-cathode discharge for emission spectrographic purposes has been known for some time. In the past it has been used primarily for theoretical and research problems involving the study of rare earth spectra *(2)*, hyperfine structure in line spectra *(3-6)*, isotopic analysis *(7-9)*, term analysis of AmI and AmII spectra *(10)*, and nuclear spin of actinium 227 *(11)*. Only rerecently has it been applied to analytical problems. McNally and co-workers *(12)* as well as Frisch and Korolov *(13)* and Rosen *(14)* determined alkali and halogen elements, while Korovin and Lipis *(15)* used it for the determination of impurities in zirconium oxide, and Birks *(16)* applied it to the analysis of samples in the form of powdered solids and solutions after they were dried in the cathode.

Brody, *et al.* *(7)* Fred and Tomkins *(10)* have used the Schüler-Gollnow, water-cooled hollow-cathode discharge *(2)* and were successful in exciting reproducibly approximately 1 mg of lithium, a light element, and neptunium, a heavy element, respectively, for several hrs, obtaining strong sharp spectral lines. Even though the tubes were operated for many hrs, it was observed that essentially all of the sample was confined to the centers of the cathode and anode, making it convenient to recover the sample.

Radiochemical examinations of the Schüler-Gollnow tube at this laboratory, after use with plutonium metal and plutonium nitrate samples, revealed that the radioactivity was confined to the anode-cathode interface. The activity found on the outside walls of the cathode was negligibly small, of the order of a few cpm. After disassembling the tube, no radioactivity was detected on the metal surfaces except at the interface.

In view of the Schüler-Gollnow, water-cooled hollow-cathode tube properties just mentioned and because of the convenience and experience already gained at this laboratory, it was decided to investigate its use in the analysis of unirradiated uranium alloys as a standin for irradiated uranium alloys. A number of uranium alloys containing approximately 2.5% Mo, 2.0% Ru, 0.3% Rh, and 0.2% Pd were investigated first. It was decided to work with unirradiated alloys initially since they were easier to handle; consequently a good deal of useful experience and spectral data could be obtained rapidly and applied to the analysis of irradiated uranium alloys later. A description of the attempt to use the Schüler-Gollnow, water-cooled hollow-cathode tube as an analytical source follows.

Experimental

Standards

The spectrographic standards used for this procedure were prepared by the Metallurgy Division of The Argonne National Laboratory. The technique employed in producing this series of uranium alloys involved the compounding of the highest composition first by induction melting in a vacuum of the order of 5 x 10^{-5} mm Hg. High purity magnesia was used for the crucible and stopper rods. The melt was held at 1450°C for ½-¾ hr before being bottom poured at 1400°C and cast in a thoria-coated graphite mold. This alloy then served as the starting material for the fabrication of all lower standards. The actual standard specimens were injection cast as rods, 0.144 in. diam., 14½ in. long. These rods were analyzed chemically, and then analytical curves plotted using an ac spark and the point-to-plane technique. For the hollow-cathode work, 5 mg of finely divided chips were taken from these rods and used in the cathode crater. The fabrication of these homogeneous standards proved to be a significant part of the spectrographic work, since the point-to-plane technique as well as the hollow-cathode method utilized small amounts of sample to plot accurate analytical curves.

Hollow-Cathode Procedure

The Schüller-Gollnow, water-cooled hollow-cathode tube is also described by Tolansky *(4)*. The anode and cathode used for the analysis of "fissium" alloys were made of 2S aluminum, and the spacing between them was maintained at 1 mm. Instead of using wax seals at the ends of the glass tube that separates the cathode from the anode, O-ring seals were employed. The connection of the hollow-cathode tube to the circulating system was made through O-ring seals and Sylphon bellows.

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*Based on work performed under the auspices of the U. S. Atomic Energy Commission.

Since very little work has been done using the hollow-cathode discharge source for the analysis of uranium alloys, it was decided to operate the hollow-cathode tube for the most part using techniques previously employed by Brody, Fred, and Tomkins of this laboratory for isotopic analysis *(7)* and hyperfine structure studies *(5,6)*. To obtain the spectrum of a uranium alloy, a 5 mg sample in the form of finely divided chips was placed in the bottom of a cathode crater. Thereafter the cathode electrode containing the sample was carefully inserted, past the ground glass joints (previously greased with Apiezon T) of the hollow-cathode tube, into the cathode piece, which was water-cooled along with the anode. The tube then was pumped out. The sample in the cathode was first operated in argon at a pressure of 3 mm Hg for 15 min for the purpose of sputtering it in the cathode crater and removing the oxide coating *(4)*. Subsequently the tube was pumped out and recharged with neon also at 3 mm Hg. The tube was then operated for 20 min to reach equilibrium conditions before exposures were made. The working tube voltage varied between 150 and 300 V, and the current was maintained at 80 ma. New cathode electrodes were used for every sample analyzed; however, one anode was used over again for approximatly 25 samples, and no cross-contamination was detected.

TABLE I. APPARATUS AND SPECTROGRAPHIC OPERATING CONDITIONS[a]

Spectrograph	Jarrell-Ash, 21 ft Wadsworth mounting, 15000 lines/in. grating, second order
Wavelength range, A	To include 3100 to 3700
Slit width, μ	25
Sector	None
Filter	None
Photographic emulsion	Eastman SA-2
Developer	Kodak D-19, 19°C, 4 min
Plate calibration	Bausch and Lomb rotating step sector and dc iron arc

[a]For the analysis of U^{235} in "fissium" alloy substitute Eastman SA-1 photographic plates and the third order of Jarrell-Ash spectrograph.

Table I shows the apparatus and spectrographic operating conditions used for the analysis of uranium alloys. The Jarrell-Ash grating spectrograph employed had enough dispersion and resolving power at 3400A in the second order to minimize the mutual interferences of the spectral lines of uranium, alloying elements, and neon, the carrier gas. A Jarrell-Ash microphotometer was used to measure the relative transmittance of the spectral lines and their adjacent backgrounds. Intensity ratios of analytical and uranium lines were obtained using a Respectra calculating board *(17)*.

Although the hollow-cathode spectrum of a uranium alloy obtained using the above procedure had many sharp lines and was simpler than the dc or ac spark spectrum in the wavelength region used, it was nevertheless complex. Hence much care had to be exercised in selecting analytical and uranium internal standard lines free of interferences for analysis and in obtaining intensity ratios of analytical lines and uranium internal standard lines to plot accurate analytical curves.

The lines shown in Table II were chosen for this work because they had no apparent interferences, optimum intensities for the nominal composition, nearly similar background for each line pair, and were in the range of approximately 600 A; hence, they were photographed in one exposure, thus expediting the spectrographic procedure.

TABLE II. WAVELENGTHS OF ANALYTICAL AND INTERNAL STANDARD LINES

Analytical lines, A	*Uranium lines, A*
Mo—3193.98	3354.50
Ru—3498.94	3493.99
Rh—3657.99	3657.82
Pd—3634.68	3636.31

Since a number of uranium lines, shown in Table II, were used for this method having background and line intensity nearly equal to the analytical line intensity for the specified composition, the errors arising from variations in attendant background *(18)* as well as plate calibration and sensitivity were minimized. As a result, accurate intensity ratios of analytical and uranium lines were obtained.

The vacuum system used for circulating the carrier gas was of conventional design and is shown in Figure 1. The unit was mounted on a wooden base approximately 2 ft by 1½ ft using a framework of aluminum rods, and it is 2 ft high. The argon and neon used in this system were supplied in 1 *l* bulbs purchased from the Linde Air Products Co. No spectral interference in the wavelength range used had been experienced due to impurities in these gases or Apiezon grease used on the stopcocks; hence traps other than liquid nitrogen were not necessary. The carrier gas was circulated by a Pyrex glass mercury vapor pump and the system was exhausted using a commercial rotary vacuum oil pump connected to the high pressure side of the diffusion pump. The direction of the gas flow shown in Figure 1 was chosen in order to provide a positive pressure at the cathode face which prevented the diffusion of the sample out of the discharge zone. The dc power supply used was of commercial design employing two 866A rectifier tubes. It had an open-circuit voltage of 1000 V and a maximum current output of 150 ma.

Results

To test the behavior of the analytical and uranium lines shown in Table II using the Schüler-Gollnow, water-cooled hollow-cathode tube, as described above, moving plate studies were made by exposing a sample for 200 min to obtain ten consecutive exposures. Ratios of the analytical and uranium lines were calculated and plotted against exposure time. Figure 2 shows the results of the test.

It is evident from the time study curves in Fig. 2 that 20 min are required for the spectral lines to give reproducible results. The six-20 min exposures taken after the 20 min "warm-up" period indicated that the analytical

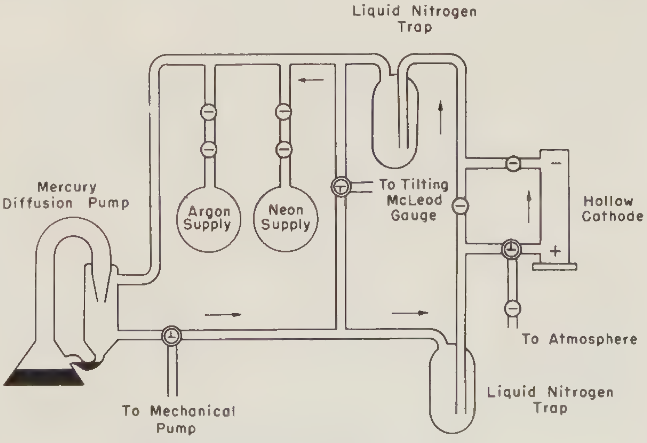

FIG. 1. CIRCULATING SYSTEM

line pairs appear stable during the two hr interval over the normally anticipated dc source variation mentioned earlier. The % std. dev. from the mean for 6 exposures for Mo was ±2.5, Ru ±5.9, Rh ±4.3, and Pd ±3.1.

Further moving plate studies made with the tube using currents of 40 and 120 ma while keeping the neon pressure constant at 3 mm Hg showed the "warm-up" period was not shortened. At a current of 40 ma, two hrs were required for the tube to reach equilibrium, while at a current of 120 ma, the tube did not reach equilibrium even when operated for three hrs.

The working curves for Mo and Ru, Rh and Pd are shown in Figures 3 and 4 respectively. They are analytical curves of wide range *(19)* and appear normal *(18)*.

Table III shows the comparison of Mo, Ru, Rh, and Pd results obtained on 6 uranium alloy samples using the Schüler-Gollnow hollow-cathode tube and the point-to-plane technique. The first three samples contain normal uranium, and the last three are approximately 50% enriched. All hollow-cathode results are the average of three determinations obtained from three separate exposures taken from one continuous 60 min excitation of the sample in the discharge tube, after the sample was operated in argon for 15 min and pre-exposed with neon for 20 min as described earlier. The results for the point-to-plane technique are also averages of three determinations. The % std. rel. dev. for the hollow-cathode results from the point-to-plane results for Mo was ±7.8; for Ru ± 5.9; for Rh ±5.4 and for Pd ±9.1.

Since the stability of the hollow-cathode discharge was affected by the size and geometry of the uranium alloy sample in the crater of the cathode, the 5 mg samples and standards used to obtain the results shown in Table II were in the form of finely divided chips, because this type of metal charge could be easily reproduced. A comparison of triplicate 20 min exposures obtained for the samples and standards showed the backgrounds and uranium line intensities were nearly the same, indicating that the discharge was stable during the interval exposures were taken. Hence, as a result of reproducible exposures, numerous photographic errors mentioned previously were minimized.

A limited amount of work was done in determining the U^{235} isotope in uranium alloy samples. Using the techniques mentioned earlier for the analysis of alloying elements in the "fissium" alloy and the apparatus and operating conditions shown in Table I, the last three samples in Table III were analyzed for U^{235}. The results obtained agreed within ±2% of mass spectrometric values.

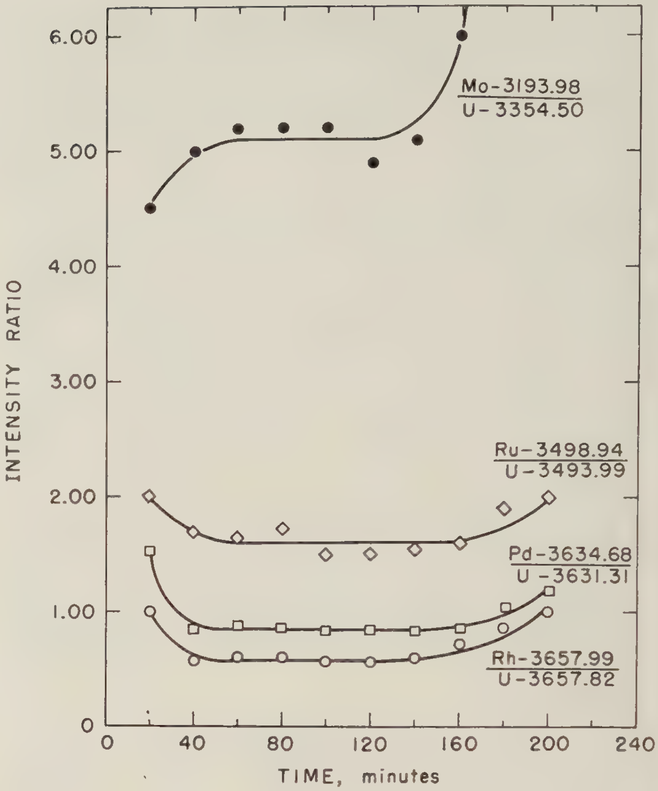

FIG. 2. TIME STUDY CURVES

Discussion

The results of this method indicate that the Schüler-Gollnow, water-cooled hollow-cathode tube lends itself readily to the analysis of uranium alloys. It has been shown that noble metals having very high melting points vaporize and diffuse slowly in the cathode crater; as a result a constant and reproducible light source is obtained, making it possible to plot accurate analytical curves. Also, it appears that the properties of the discharge, as shown by the moving plate studies are such that its field of application can be extended to the analysis of irradiated uranium alloys.

It had been observed that the use of argon instead of neon, as a carrier gas for the first 15 min, to sputter the "fissium" alloy on the wall of the cathode crater, did not appear to be critical when the metal was relatively free of oxide. However, when oxide was present in the finely divided sample, the heavier gas, argon, sputtered the sample on the wall of the cathode crater faster, and as a result more

TABLE III. COMPARISON OF HOLLOW-CATHODE AND POINT-TO-PLANE RESULTS[a]

Sample No.	*Mo%*				*Ru%*				*Rh%*				*Pd%*			
	H.C.	*P-t-P*	*Diff.*	*% Rel. Dev.*	*H.C.*	*P-t-P*	*Diff.*	*% Rel. Dev.*	*H.C.*	*P-t-P*	*Diff.*	*% Rel. Dev.*	*H.C.*	*P-t-P*	*Diff.*	*% Rel. Dev.*
36748	2.47	2.62	0.15	5.7	2.25	2.20	0.05	2.3	0.290	0.286	.004	1.4	0.210	0.215	.005	2.3
36749	2.52	2.42	0.10	4.1	2.00	2.05	0.05	2.4	0.245	0.265	.020	7.5	0.194	0.192	.002	1.0
36750	2.44	2.58	0.14	5.4	1.98	2.14	0.16	7.5	0.255	0.270	.015	5.6	0.188	0.206	.018	8.7
37659	2.31	2.19	0.12	5.5	1.88	2.00	0.12	6.0	0.230	0.245	.015	6.1	0.170	0.196	.026	13.3
37660	2.46	2.19	0.27	12.3	2.00	1.86	0.14	7.5	0.255	0.255	.000	0.0	0.210	0.203	.007	3.5
37661	2.42	2.27	0.15	6.6	1.95	2.02	0.07	3.5	0.235	0.245	.010	4.1	0.185	0.210	.025	11.9
Std. % Rel. Dev.				±7.8				±5.4				±5.9				±9.1

[a] % Rel. dev. expressed as deviation of hollow-cathode results from point-to-plane results, H.C.—Hollow-cathode method, P-t-P—Point-to-plane method.

intense spectra of the alloy were obtained quicker. Because it was possible that "fissium" samples submitted for analysis might contain some oxide, it was decided to use argon first to sputter the sample on the cathode crater wall instead of neon; as a result, uniform exposures of the alloy were obtained, and the spectrographic procedure was expedited, since the use of argon contributed to more intense spectra faster. To minimize the mutual inteferences between the spectral lines of the carrier gas and "fissium" alloy, neon was used instead of argon to excite the sample to obtain the spectrum of the alloy, since the former had somewhat less background associated with its spectrum in the wavelength region used.

It is thought that the Schüler-Gollnow, water-cooled hollow-cathode tube might have some advantages over a hot hollow-cathode *(15)* for the analysis of uranium alloys, since the former minimizes the mutual interferences between uranium and analytical lines caused by background and broad lines; hence more suitable lines for densitometry are to be expected. A further advantage of the Schüler-Gollnow, water-cooled hollow-cathode tube over the hot hollow-cathode tube for the analysis of uranium alloys was realized when the former was dismantled and examined for radioactivity, after being used to analyze the samples shown in Table III and standards. The radiochemical examinations of the water-cooled aluminum electrodes and metal surfaces of the tube indicated the radioactivity was confined to the same areas of the tube as previously discussed for plutonium, thus making it convenient to recover a sample. This is an important and critical point in attempting to develop a spectrographic method for the analysis of highly radioactive samples, and it confirms the findings of Newbound and Fish *(20)*, who recovered a majority of an unspecified radioactive material after operating a water-cooled hollow-cathode tube for 8 or 10 hrs.

Much of the success obtained with this method using a Schüler-Gollnow, water-cooled hollow-cathode can be attributed to an efficient vacuum system. After the tube was open to atmospheric pressure, and loaded with a sample, approximately 5 min was required for the system to be pumped out, carrier gas added, and discharge started. The system was sufficiently vacuum tight so that no instability of the discharge due to impurity gases were observed; as a result reproducible spectra were obtained. After several months of using this circulating system, only insignificant amounts of mercury from the diffusion pump were observed in the hollow-cathode spectrum of the uranium alloy, indicating properly located and effective liquid nitrogen cold traps.

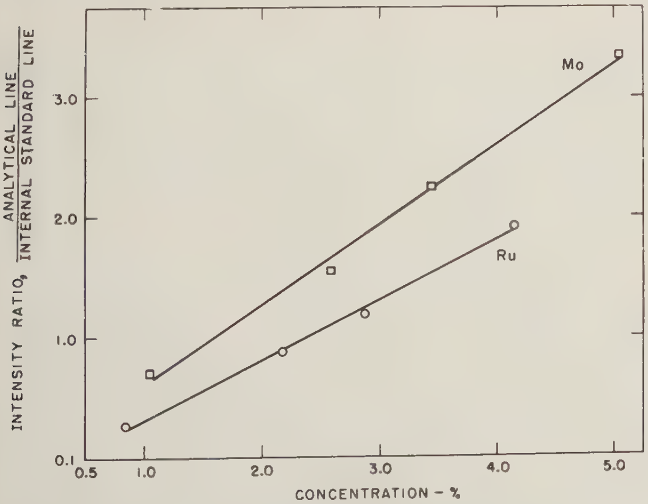

Fig. 3. Working Curves for Molybdenum and Ruthenium

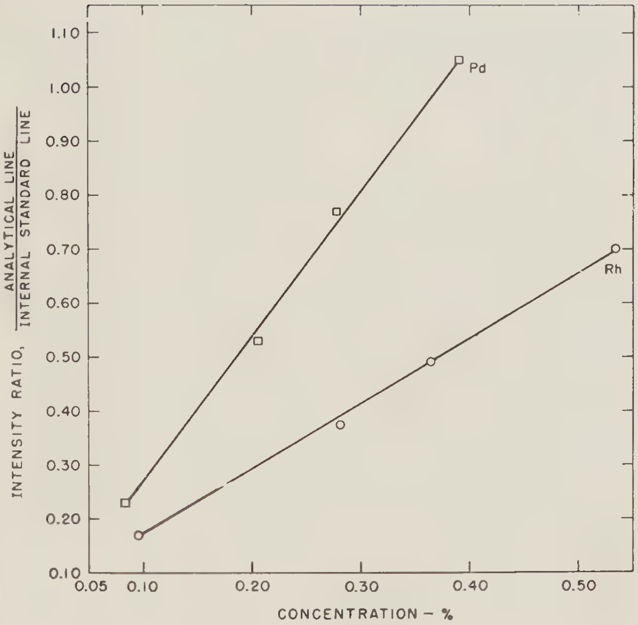

Fig. 4. Working Curves for Rhodium and Palladium

It was decided to include the working curves in this paper (Figures 3 and 4) because very few of them have been drawn and illustrated in the past using the hollow-cathode tube as an analytical source, and also because they appear to be normal curves, which is not always obtained in optical emission spectrographic work. Experience with the analytical curves shown in this paper proved they were reproducible, and the results obtained from them were in good agreement with the point-to-plane technique.

Since this spectrographic procedure employs a relatively new technique to analyze uranium alloys, it might be of some interest to point out the time spent to analyze the samples shown in Table III. Approximately 24 man-hrs were needed to run the standards and samples. However, this time is still appreciably faster than the time required by routine chemical methods *(21,22)* for the determination of alloying constituents in uranium metal, and it might be reduced considerably by using faster photographic plates or photoelectric recorders.

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The Application of Statistical Methods to a Problem In Spectrochemical Analysis[*]

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Abstract

An $\overline{R}$ chart, analysis of means charts, and the range square ratio test, are applied to spectrographic data to compare two internal standards and two different buffer salts in the analysis of solutions of organic ash. The range square ratio test confirms the indications derived from the graphical methods that cobalt is a better standard for manganese, molybdenum is better for lead, and that sodium silicate is superior to lithium chloride as a buffer salt.

Introduction

The increasing number of papers published during the past few years on the application of mathematical and statistical methods to the analysis of spectrographic data indicates a growing acceptance of these techniques among spectrographers. A paper by Nelson (*1*) explains the use of some statistical methods in terms understandable to spectrographers with no background in statistics. He also gives a review of recent literature on these applications. Strasheim and Keddy (*2*) explain the use of scatter diagrams in the preparation of analytical curves. Shewell makes the point strongly in his paper (*3*).

The purpose of the experiment reported here is to determine whether Co or Mo is better as a standard for the analysis of Pb, Mn, and Cu, and to test the effectiveness of LiCl *vs.* Na_2SiO_3 as buffer salt in the presence of varying amounts of Na, Ca, K, and Sr.

Quantitative spectrochemical analysis is based on measurement of the intensity of a spectral line emitted by an element when it is excited by an electrical discharge, as in a high voltage arc. In the photographic method used here, the procedure is subject to variability of two general types. One may be considered as random error and is due to such things as: slight variation in arc current during excitation, in development of the photographic plates, or in densitometry of the spectral lines. In the analysis of the ash of organic materials, a second, systematic, and more serious cause of error lies in variable composition of the ash with regard to alkalies. This may have a marked effect on the intensity of the spectral lines of the heavy metals for which the sample is being analyzed.

Errors due to the first, or random, type of variation may be effectively reduced by the use of an internal standard which, ideally, will react to variations inherent in the method in a manner similar to that of the elements being measured. The presence of alkalies, however, may affect the spectral lines of the analysis element and standard element to a different extent, thus adding the second source of error mentioned above.

In preparing standard curves for use in analyzing organic materials, the synthetic standards should contain not only the analysis element and standard element, but should simulate as closely as possible the physical characteristics and chemical composition of samples for which the standard curves are to be used. The problem of duplicating physical form and insuring homogeneity of samples and standards may be met by ashing organic samples and dissolving the ash in dilute nitric acid. Standards may then conveniently and accurately be prepared as solutions. In a laboratory handling non-routine samples from many different sources, it is not feasible to prepare standards to

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TABLE I. PLAN OF EXPERIMENT

		Standard I				Standard II			
Buffer		Lithium Chloride				Sodium Silicate			
Alkali additions		1 Ca	2 Na	3 K	4 Sr	5 Ca	6 Na	7 K	8 Sr
Concn. of alkalies, %	1—	0.2				0.2			
	2—	0.5				0.5			
	3—	1.0				1.0			

fit each sample, even if the alkali content of each were known in advance. Compensation for differences in alkali content of the ash of different samples may be partially achieved by adding an excess of some "buffer" salt to both standard and sample solutions. The effect of the buffer is to suppress variation in spectral line intensity due to difference in sample composition.

The accuracy of the spectrochemical method for analysis of samples of variable composition is largely dependent on the proper choice of internal standard elements and selection of an efficient buffer material. Consideration of physical characteristics of metals such as volatility, excitation potentials of usable spectral lines, and proximity in the periodic table help in making a first guess as to which ones may be appropriate standards for others. The final choice must be made by trial of different combinations under conditions representing normal variations in operating procedure. Thus, different buffer salts must be investigated to discover which ones will provide the most stable ratio of analysis line to standard line in the presence of the variation in ash composition to be expected in samples to be analyzed.

Experimental

Preparation of Solutions

A solution in 10% HNO_3 was made containing the following as analysis elements: 0.001% Fe, 0.001% Pb, 0.001% Mn, and 0.0005% Cu, and as internal standard elements, 0.01% Mo and 0.01% Co. Two standard solutions were prepared. Standard I was made by adding LiCl to the above solution to make it 1% in LiCl, and for Standard II, Na_2SiO_3 was added to make it 1% in Na_2SiO_3. Standards I and II were further subdivided by adding, to different aliquots, Ca, Na, K, and Sr, to provide three different levels of concentration, 0.2%, 0.5%, and 1.0% of each alkali. There were 24 solutions, from each of which three spectrograms were taken. The plan of the experiment may be represented as shown in Table I.

Spectrographic Procedure

The apparatus and conditions are shown in Table II. From each of the 24 solutions, 3 pairs of electrodes were prepared by evaporating 0.05 ml of solution on the flat surface of each electrode. Twelve exposures were made on each of six plates. Since some variation between plates is expected, the order of the individual runs was randomized using a table of random numbers. The ratio of intensity of analysis line to standard line was calculated for each pair under investigation. In most cases the most intense spectral line of the analysis element was used, and an internal standard line near the same wavelength was chosen to avoid differences due to emulsion characteristics and background in different wavelength regions. The selected lines are given in Table III.

TABLE II. APPARATUS AND SPECTROGRAPHIC OPERATING CONDITIONS

Electrodes	Graphite rods, 3/8 in. diam. sealed with collodion solution
Analytical gap, mm	2
Excitation Unit	National Spectrographic Laboratories Uniarc
Capacitance, μfd	0.0025
Inductance, μh	0
Current, r.f. amp	4
Discharges per half-cycle	2
Spectrograph	Bausch and Lomb medium prism
Wavelength range, A	2200 to 4500
Sector	None
Filter	None
Photographic emulsion	Eastman SA-1
Developer	Kodak D-19, 20° C, 3 min
Plate calibration	Two line method, iron arc exposure
Densitometer	National Spectrographic Laboratories

TABLE III. ANALYTICAL LINES

Analysis Element Element	Analysis Element Wavelength, A	Standard Element [a] Element	Standard Element [a] Wavelength, A
Fe	2599	Co	2580
Mn	2606	Co	2580
Mn	2606	Mo	2684
Pb	2833	Co	2580
Pb	2833	Mo	2816
Cu	3274	Co	3414
Cu	3274	Mo	3194

[a] Molybdenum was not used as a standard for iron because a previous experiment had indicated the superiority of cobalt.

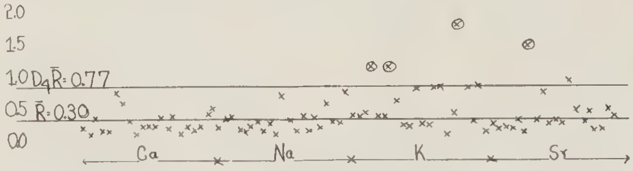
Statistical Techniques Used

Two techniques which display the data in graphical form were chosen, since this type of presentation is readily understood by people without an extensive background in statistics. The conclusions indicated by these were confirmed by application of a particular form of the F test for difference in variance. These techniques, all of which use the range in the estimation of variance, are simpler, for a large amount of data, than are those employing the sums of squares of deviation from the mean for calculation of variance.

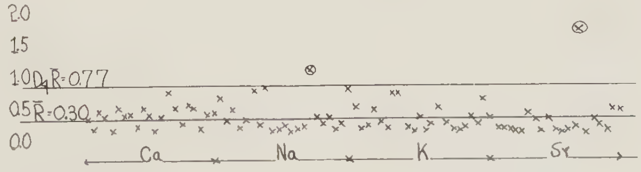
Results and Discussion

$\bar{R}$ Chart

The points plotted are ranges (maximum minus minimum) of all analysis element to internal standard intensity ratios for the three photographs from each solution (Figure 1). These are assumed to represent the random variation inherent in the method, including differences between photographic plates. This chart spots outliers, which were discarded, and gives an indication of reproducibility under the various conditions of the experiment.



LiThium Chloride



Sodium SilicaTe

FIG. 1. $\bar{R}$ CHART

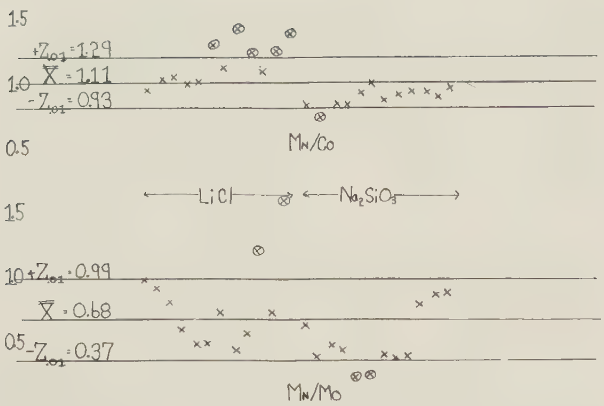


FIG. 2. ANALYSIS OF MEANS CHART FOR MANGANESE

There are 168 points, including 12 each for Fe/Co, Mn/Co, Mn/Mo, Pb/Co, Pb/Mo, Cu/Co, and Cu/Mo in the LiCl buffer and 12 each in the Na_2SiO_3 buffer. The upper control limit *(4)*, D_4R, represents the value below which 99.7% of the ranges will fall if random variation only is affecting the result. The range chart shows six points which are far above the control limit. Reference to the original data indicated that, in each case, one of the three ratios was far out of line. This effect is usually due to non-uniform deposit on the electrode or poor drying characteristic of the solution. It is noteworthy that four of the six were found in the LiCl buffer. Visual inspection of the range chart also indicates somewhat smaller values in Na_2SiO_3 buffer when all alkali additions are considered, although LiCl is better with Ca and Na.

Analysis of Means *(5)*

These charts indicate which of the two internal standard elements used, (Co or Mo) gives the most constant ratio when used with Mn, Cu, and Pb, respectively (Figures 2, 3, and 4).

The points plotted are the average intensity ratios for the three photographic replicates from each solution. The individual averages are compared, in each case, with the grand average or group mean. If individual averages fall outside the decision lines, we may say there is evidence of non-random variation, with a risk of 1% that there is, in fact, no assignable difference among the means. These decision lines are drawn at levels represented by:

$$\bar{\bar{X}} \pm Z_\alpha(k,\partial)\sigma_{\bar{x}}$$

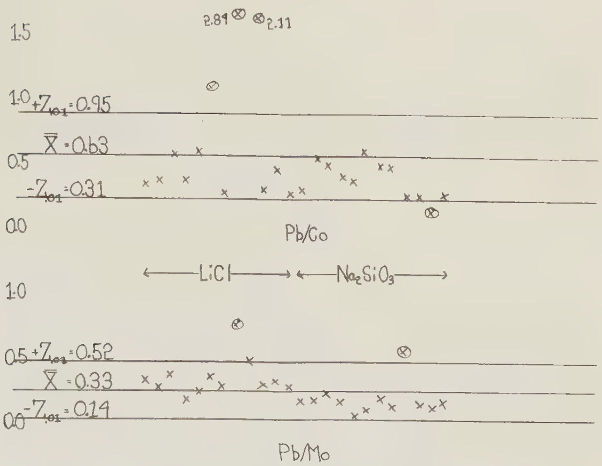


FIG. 3. ANALYSIS OF MEANS CHART FOR LEAD

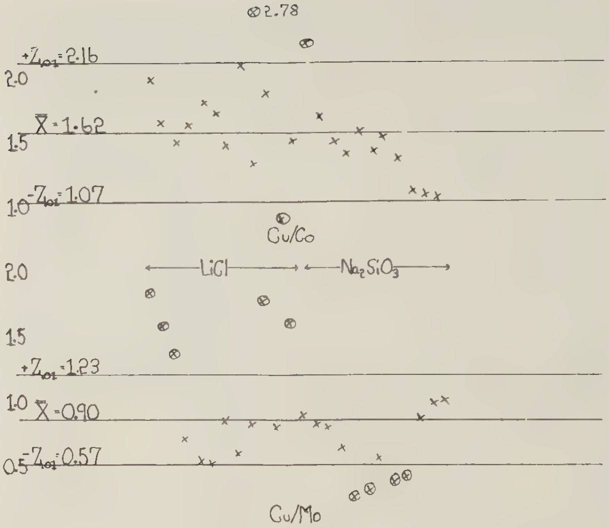


FIG. 4. ANALYSIS OF MEANS CHART FOR COPPER

where: $\bar{\bar{X}}$ is the grand mean, Z (3.73) (from Table 2-B of the reference cited above (5)) is dependent upon: α = risk = 1%, k = number of means = 24, ∂ = 0.9k(n−1) = 0.9 (24)(2) = 43 degrees of freedom for k means of sample size of n = 3, and $\sigma_{\bar{x}} = \sigma/n^{1/2} = \sigma/3^{1/2}$; $\sigma = \bar{R}/d_2$ *(4)*.

$\sigma_{\bar{x}}$ is the standard deviation of the mean calculated from the average range, $\bar{R}$; that is, the average of the ranges observed for the three replicates of each respective analytical line to standard line intensity ratio in all runs, including both buffers. Calculations are shown in Table IV.

All analysis of means charts show a number of points outside the decision lines, indicating variability greater than that represented by the replicate photographs. Examination of the charts, however, indicates the following:

1) The averages for Mn/Co in Na_2SiO_3 buffer are shifted to a lower level than in LiCl buffer and are much more constant than Mn/Co in LiCl, or Mn/Mo in either buffer. Cobalt is indicated as the better standard element, and Na_2SiO_3 as the better buffer.

2) It is not apparent which buffer is better for Pb, but the dispersion is smaller using Mo as standard in both buffers.

3) The results seem poor for copper with either standard, although all points except one are within the decision lines using cobalt as standard in Na_2SiO_3 buffer. The wide spread of the decision lines from the mean indicates poor reproducibility in both cases. Further trial of other standards is indicated for copper.

TABLE IV. CALCULATIONS FOR ANALYSIS OF MEANS CHARTS[a]

Fig.	*Ratio*	$\bar{\bar{X}}$	$\bar{R}$	σ	$\sigma_{\bar{x}}$	$\bar{\bar{X}}-Z\sigma_{\bar{x}}$	$\bar{\bar{X}}+Z\sigma_{\bar{x}}$
2	Mn/Co	1.11	0.14	0.083	0.048	0.93	1.29
	Mn/Mo	0.68	0.24	0.142	0.082	0.37	0.99
3	Pb/Co	0.63	0.25	0.148	0.085	0.31	0.95
	Pb/Mo	0.33	0.15	0.089	0.051	0.14	0.52
4	Cu/Co	1.62	0.43	0.254	0.147	1.07	2.16
	Cu/Mo	0.90	0.26	0.153	0.088	0.57	1.23

[a] Z = 3.73 (Table 2-b, (5))

TABLE V. RANGE SQUARE RATIO FOR INTERNAL STANDARDS

$\bar{R}$		F_R [a]
Mn/Co 0.14	Mn/Mo 0.24	$\frac{0.24^2}{0.14^2} = 2.94$
Pb/Co 0.25	Pb/Mo 0.15	$\frac{0.25^2}{0.15^2} = 2.78$

[a] $F_R = \bar{R}_1^2/\bar{R}_2^2$ (1a, (6))

Range square ratio test of Duncan *(6)*

The analysis of means charts display the intensity ratios found and indicate in which combination of internal standard and buffer they are most constant for each analysis element. It is also desirable to know under which conditions the reproducibility of replicate runs is best, or in other words, in which case the standard deviation is smallest. The spread of the decision lines on the analysis of means charts, which are proportional to the standard deviation, indicate that cobalt is a better standard for manganese, and that molybdenum is better for lead. The range chart indicates that fewer points are beyond the control limits in the Na_2SiO_3 buffer than with LiCl. In order to determine whether the differences indicated by the charts represent a real difference in variability, the range square ratio test was used.

This method uses the F test to compare ranges of replicate runs for significant difference, which, if found, indicates a real difference in variance. The ranges found for Mn and Pb, using each of the two standard elements, and including values found in both buffers, were compared. Results are shown in Table V. The F ratio above which a significant difference may be assumed to exist, at the 99% level of confidence, for 44 degrees of freedom in each variance estimate, is about 2.1. Since all experimental F ratios are above this figure, there is evidence that Co is a better standard for Mn, and Mo is better for Pb. This confirms the evidence of the analysis of means charts in which the respective separation of the decision lines give the same indication.

The variances found for the ratios Mn/Co, Pb/Mo, and Fe/Co were then compared in the two different buffers, again using the range square ratio test, with the results shown in Table VI. There are 22 degrees of freedom for these estimates. The critical F ratios in this case are 2.80 at the 99% level of confidence, and 1.75 at the 90% level. Thus, there is evidence that Na_2SiO_3 is definitely better for Pb/Mo, slightly better for Mn/Co, and there is no real difference for Fe/Co.

TABLE VI. RANGE SQUARE RATIO FOR BUFFERS

	$\bar{R}$ LiCl	Na_2SiO_3	F_R [a]
Mn/Co	0.16	0.12	$\frac{0.16^2}{0.12^2} = 1.78$
Pb/Mo	0.19	0.10	$\frac{0.19^2}{0.10^2} = 3.61$
Fe/Co	0.26	0.31	$\frac{0.31^2}{0.26^2} = 1.42$

[a] $F_R = \bar{R}_1^2/\bar{R}_2^2$ (1a, (6))

Conclusions

The conclusions to be drawn from the statistical analyses used may be summarized as follows:

1) The range chart (Figure 1) shows more points outside of control limits with the LiCl, than with the Na_2SiO_3 buffer.

2) The analysis of means charts (Figures 2, 3, and 4) confirm the observation that Na_2SiO_3 is preferable as buffer for manganese; and further indicate the superiority of cobalt as the standard for manganese, and molybdenum for lead.

3) The range square ratio test gives the same information concerning respective standards for manganese and lead, and also indicates the general superiority of Na_2SiO_3 buffer which was found by the range chart.

Acknowledgment

The author gratefully acknowledges helpful suggestions made by Dr. Ellis R. Ott, Director of the Statistics Center, Rutgers University

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Infrared Spectra of Polynuclear Aromatic Compounds in the C-H Stretching and Out-of-Plane Bending Regions*

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Abstract

A study has been made of the infrared spectra of 33 poly-nuclear aromatic compounds including some known carcinogens. In most cases, the number of fused rings may be determined by measuring the frequency of the characteristic aromatic (C-H) stretching vibration with a LiF prism. For unsubstituted fused rings, the (C-H) stretching vibration shifts to lower frequencies as the number of fused rings increases. In addition, the position of the fused rings can be established from data obtained from the out-of-plane (C-H) bending vibrations in the range of 700 to 900 cm^{-1}.

Introduction

The identification of polynuclear aromatic compounds poses an interesting problem to the analytical chemist. These substances are encountered in many fields, for example, in air pollution studies, in pyrolysis products, and in the petroleum industry. Because certain members are carcinogenic, the determination of the discrete individuals comprising a mixture becomes a pressing problem. Separations often involve chromatographic techniques, while the main methods of identification include mass spectrometry, and ultraviolet and infrared spectroscopy.

In 1949, Pacault and Lecomte *(1)* searched for an infrared absorption band characteristic of cancer-producing compounds but with a negative result. In the following year, Thompson and co-workers *(2,3)* studied the infrared spectra of some known carcinogens with emphasis on derivatives of stilbene, anthracene, 1,2-benzanthracene, and dibenzanthracene.

Subsequently, Cannon and Sutherland *(4)* reported the spectra of some 104 aromatic compounds including many polynuclear aromatic compounds with major emphasis on the C-H out-of-plane deformation region. Fuson and Josien *(5)* studied the spectra of derivatives of 1,2-benzanthracene and stated that the aromatic stretching frequency at approximately 3050 cm^{-1} showed more correlation with carcinogenic activity and with indices of electronic structure than any other infrared band. In previous studies the spectra were obtained on the compounds dissolved in such solvents as carbon disulfide, carbon tetrachloride, or chloroform, or Nujol mulls. In this investigation, the polynuclear aromatics were studied in the solid phase in potassium bromide. Two main regions were emphasized, the C-H stretching region from 2700 to 3100 cm^{-1} and the out-of-plane C-H bending vibrations from 700 to 900 cm^{-1}.

Experimental

Most of the compounds were Eastman Kodak, White Label grade, and some were A.P.I. samples of better than 99% purity. In many cases, the ultraviolet spectra revealed no traces of impurities. The infrared spectra were measured on a Perkin-Elmer Model 21 and also on a Perkin-Elmer Model 12C recording infrared spectrometer equipped with a lithium fluoride prism. The prism was calibrated using the rotational bands of methane, hydrogen chloride, carbon monoxide, and hydrogen bromide. The wavenumber values are reproducible to a precision of 1 cm^{-1} and to an accuracy of 2 or 3 cm^{-1}. Daily measurements on hydrogen chloride gas were made as a routine check on the wavelength calibration.

Results and Discussion

The 2800-3100 cm^{-1} Region

The absorption bands obtained in the C-H stretching region for the compounds under consideration are tabulated in Table I. In a series of symmetrical compounds such as naphthalene, anthracene, napthacene, and pentacene, the first two members absorb at the same frequency (3051 cm^{-1}),' while naphthacene and pentacene absorb at 3044 and 3039 cm^{-1}. Thus, in unsubstituted compounds the main absorption band shifts to lower frequencies as the number of fused rings increases. Simplification of the spectra is observed in this series as the number of fused rings increases, with naphthalene producing six bands in the 2965-3081 cm^{-1} region as compared to only three for pentacene.

In the case of compounds containing two fused rings, when substitution occurs in the 1-position the aromatic C-H stretching vibration appears as a doublet usually at 3044 to 3062 cm^{-1} (Figure 1). Two additional bands at 3008 and 3096 cm^{-1} are remarkably constant for these compounds. Only one peak is observed at 3051 cm^{-1}, similar to naphthalene, when substituents are in other than the 1-position. When one or more methyl groups are directly attached to a fused ring, four or five bands are observed in the region 2850-3000 cm^{-1}. Similar observations for methyl substituents have been made by Fuson and Josien *(5)* for derivatives of 1,2-benzanthracene. When the methyl groups are linked to the ring by methylene groups, correlations for methyl and methylene groups similar to those for straight and branched chain hydrocarbons were observed, with the CH$_3$ bands being at 2960 and 2870 cm^{-1} and the CH$_2$ bands at 2930 and 2860 cm^{-1} *(6)*. Acenaphthene which is substituted in the 1-position likewise shows a doublet structure, with the CH$_2$ bands occurring in the normal range.

In the case of the 3-ring compounds the main band occurs at 3051 cm^{-1} regardless of whether sulphur is present in the ring.

Two signficant features were produced by 9,10 substitution of an anthracene ring: (1) Depression of the intensity of the C-H stretching vibration; and (2) a splitting of the main band into a pronounced doublet. The 9,10 substituted compounds had to be measured at a concentration as high as 8% in some cases as compared to 2% for the other compounds studied.

The 4-ring compounds show a strong band centered at 3044 cm^{-1} with chrysene (3051 cm^{-1}) being the only exception. The 5-membered rings have their main band

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*Presented at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, March, 1960.

TABLE I. CARBON-HYDROGEN STRETCHING FREQUENCIES OF POLYNUCLEAR AROMATIC COMPOUNDS

RANGES: 3 Rings (3047-3052 cm^{-1}), 4 Rings (3040-3045 cm^{-1}), 5 Rings (3033-3040 cm^{-1})

Compound	*Frequency, cm^{-1a}*												
2 FUSED RINGS													
Naphthalene							2965 w		3001 m	3028 m	3051 s	3062 m	3081 w
1-Methylnaphthalene		2860 m		2906 m	2930 m	2942 s	2969 s		3008 m		3044 s	3065 s	3096 w
1-*n*-Propylnaphthalene			2870 m		2930 s		2960 s		3005 w		3044 s	3062 s	3096 w
1-*n*-Butylnaphthalene		2858 s	2867 s		2928 s		2956 s		3005 w		3041 s	3062 s	3094 w
1-*n*-Pentylnaphthalene		2856 s	2870 s		2928 s		2956 s		3005 w		3044 s	3062 s	3096 w
Acenaphthalene	2836 s	2855 w	2870 w	2882 w	2920 s	2938 m			3012 w	3037 s	3053 s		3096 w
2-Methylnaphthalene		2858 m			2916 s	2931 w	2969 w		3017 m		3053 s		3087 w
2,3-Dimethylnaphthalene		2850 m	2878 w	2897 w	2920 m	2932 s		2980 m	3010 m		3051 s		3078 w
2-*n*-Tetradecylnaphthalene		2852 s	2869 w		2920 s		2952 m		3010 w		3051 m		3078 w
3 FUSED RINGS													
Anthracene								2986 m	3008 m	3021 m	3051 s	3089 w	3109 w
Phenanthrene									3004 m	3037 w	3053 s	3080 w	3091 w
Thianthrene							2962 m		3010 w		3047 s		3113 w
Dibenzthiophene							2960 w		3017 m		3049 s	3060 s	3111 w
9-Methylanthracene			2866 w		2924 m			2990 w	3010 w	3024 m	3051 s		3081 w
2-Methylanthracene				2912 m	2930 w		2972 w	3005 w	3021 m	3051 s		3091 w	3111 w
3 FUSED RINGS, 9,10 SUBSTITUTED													
Anthraquinone								3008 w	3035 m			3073 s	3115 w
2-Methylanthraquinone		2858 m			2920 s	2951 m	2983 m			3040 s		3065 s	
Anthrone	2773 m	2847 m	2871 s	2879 s	2920 s	2951 m		3008 m	3028 s		3058 s		3094 w
2-Methyl-1-nitroanthraquinone	2747 w			2907 m	2920 w		2962 w	3015 w		3042 m		3089 s	3120 w
9,10-Dibromoanthracene							2973 w		3033 s			3073 s	3100 w
1,2-Benzanthraquinone								3017 m			3060 s		3111 w
Phenanthrenequinone									3027 m		3065 s	3085 s	3103 w
4 FUSED RINGS													
Chrysene								3019 w	3037 w	3051 s		3080 m	3091 m
Benz(a)anthracene								3001 w	3028 m	3044 s			3098 w
Pyrene							2967 w	2990 w	3028 m	3044 s		3078 w	3098 w
Naphthacene							2975 w	3001 w		3044 s	3058 m	3078 m	3111 w
Fluoranthene							2957 w	2990 w		3044 s		3078 w	3091 w
4 FUSED RINGS, 9,10 SUBSTITUTED													
9,10-Dimethylbenz(a)anthracene	2847 w	2862 m			2916 s	2951 m	2982 m		3026 m	3042 m	3058 s		3096 w
5 FUSED RINGS													
Dibenz(a,h)anthracene								3021 m	3033 s	3051 s	3071 s		3111 w
Benzo(a)pyrene									3033 s	3046 m		3072 m	3094 w
Perylene							2958 w	3008 w		3047 s		3080 w	3098 w
Pentacene							2960 w		3039 s		3072 w		
3-Methylcholanthrene	2836 w	2852 m		2910 s	2924 s			3019 m	3037 s		3062 m		

[a] The notations, s, m, w, indicate bands of strong, moderate, and weak intensity respectively.

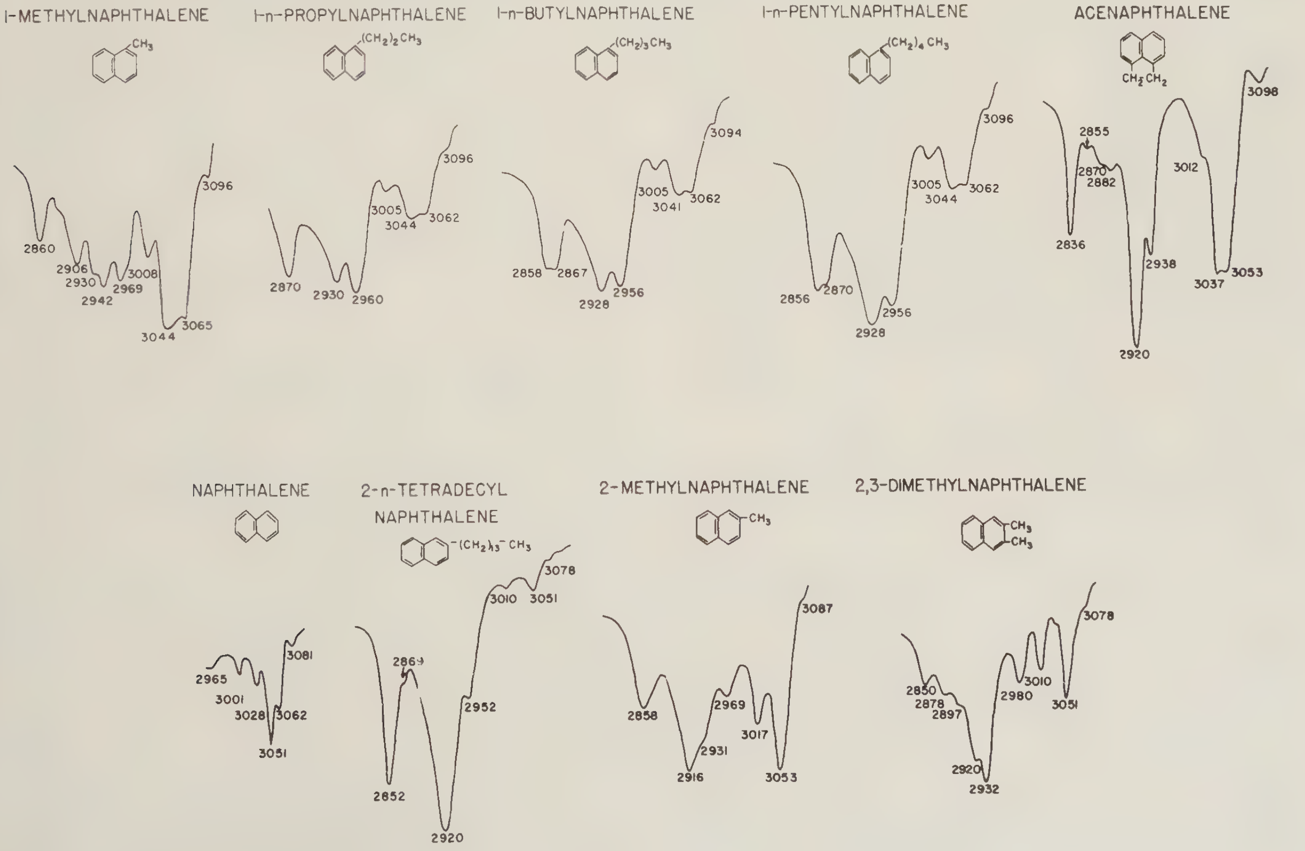

FIG. 1. INFRARED SPECTRA OF SOME TYPICAL POLYNUCLEAR AROMATIC COMPOUNDS IN THE C-H STRETCHING REGION

centered at 3037 cm^{-1} with perylene (3051 cm^{-1}) providing an exception.

The 700-900 cm^{-1} Region

Correlations for substituted benzene rings may be applied to polynuclear aromatic compounds, but the known absorption regions for the out-of-plane C-H bending vibrations must be broadened. The ranges are as follows: 4 adjacent hydrogens—(720-780 cm^{-1}), 3 adjacent hydrogens—(780-800 cm^{-1}), 2 adjacent hydrogens—(800-910 cm^{-1}), 1 hydrogen—(860-910 cm^{-1}). Although the pattern is fairly complex, these bands can be identified as the strongest in this region. Weak and medium bands are no indication as to the type of substitution.

The absorption bands of the compounds studied in this region are listed in Table II. Although these values agree reasonably with those published by Cannon and Sutherland *(4)*, there are more individual sharp bands. In many cases doublet or triplet bands in the potassium bromide pellet were obtained as compared to a single broad band in a Nujol mull or solution. The improvement in resolution and the differences observed between the solid phase and solution spectra are not unexpected *(7)*.

Anthraquinone and two of its derivatives provide the main exception to the correlation. The presence of four adjacent hydrogen atoms should cause absorption in the 750 cm^{-1} region; however, only one strong band is present at 820 cm^{-1} for anthraquinone. Although there are bands in the correct region for single and double adjacent hydrogen atoms in 2-methylanthraquinone and 1-nitro-2-methylanthraquinone, they are not of the expected intensity. Highly condensed ring systems such as pyrene (840 and 750 cm^{-1}) also fail to follow the correlation.

Naphthalenes substituted in the 1-position show a doublet at 800 and 780 cm^{-1} which can be attributed to three and four adjacent hydrogen atom vibrations. When substituents are in the 2-position, the expected band at 900 cm^{-1} for a single hydrogen atom is weak, although in 2,3-dimethylnaphthalene this band is present at 880 cm^{-1}. The 900 cm^{-1} band varies in strength with the number of single hydrogen atoms present and is very strong when they are in the 9,10-position.

In many cases the 750 cm^{-1} band was present in the form of a doublet or triplet with several side bands.

The spectra of four known carcinogens, benz(a)anthracene, dibenz(a,h)anthracene, benzo(a)pyrene and 3-methylcholanthrene show a doublet structure in the C-H stretching region, but so do many of the other compounds studied. Although their spectra in the rock salt region is complex, with more bands on the average than most of the other polynuclear compounds investigated, this fact may be explained by the unsymmetrical nature of these compounds.

Acknowledgment

This work was supported in part by the Undergraduate Research Participation Program sponsored by the National Science Foundation. The authors are indebted to

TABLE II.
CARBON-HYDROGEN OUT-OF-PLANE BENDING FREQUENCIES OF POLYNUCLEAR AROMATIC COMPOUNDS

RANGES: 4 Adjacent Hydrogens (720-780 cm^{-1}), 3 Adjacent Hydrogens (780-800 cm^{-1}), 2 Adjacent Hydrogens (800-850 cm^{-1}), 1 Single Hydrogen (860-910 cm^{-1})

Compound	*Frequency, cm^{-1a}*											
2 FUSED RINGS												
Naphthalene				845 w	820 w	790 s	780 s					
1-Methylnaphthalene				852 w			790 s	770 s		732 m		700 m
1-*n*-Propylnaphthalene			865 w	851 w		800 s	792 s	776 s	745 w	730 m		690 w
1-*n*-Butylnaphthalene				858 w		800 s	783 s	775 s	745 w	730 m	712 w	690 w
1-*n*-Pentylnaphthalene			860 m		832 w		795 s	775 s		730 m		690 w
Acenaphthalene		892 w		840 s		800 s	785 s		750 m			
2-Methylnaphthalene		890 w		851 m		818 s		768 w	741 s			702 w
2,3-Dimethylnaphthalene		872 s							742 s			
2-*n*-Tetradecylnaphthalene		890 w		851 m		816 m	795 w	775 m	741 s		720 m	
3 FUSED RINGS												
Anthracene	908 m	885 s	860 w	845 w				755 w	742 m	736 s	725 s	710 w
Phenanthrene		872 s	865 m		830 m	815 s				735 s	715 s	
Thianthrene			878 m	870 m				762 s	750 s		715 w	
Dibenzthiophene			862 w					768 w	745 s	732 s	728 m	702 m
9-Methylanthracene		890 s	860 w	840 m				778 s		730 s	720 s	
2-Methylanthracene	905 m	889 s	869 w			800 m			741 m	731 s		
3 FUSED RINGS, 9,10 SUBSTITUTED												
Anthraquinone		892 w			820 m	810 s				720 w		695 s
2-Methylanthraquinone		890 w		850 m		810 w	790 w				710 s	
Anthrone		880 w	860 w	840 w		810 s	790 m			720 m	712 s	692 m
2-Methyl-1-nitroanthraquinone		890 w	880 m	852 s	830 m		792 m	770 s	742 s	738 m	710 s	
9,10-Dibromoanthracene		890 w		845 m	832 m			756 s	742 s	728 w		
Benz(a)anthraquinone		875 m	860 m	845 s	820 m	802 m	792 m	770 s	755 s		710 s	
Phenanthrenequinone							780 m	770 s	759 s		712 s	
4 FUSED RINGS												
Chrysene	900 w	885 w	862 m			815 s		758 s	748 m		710 w	678 m
Benz(a)anthracene	900 m	888 s		847 m		815 s	783 m		749 s			690 m
Pyrene	908 w	890 w		840 s	820 m	800 w		781 m	750 s			708 s
Naphthacene	900 s	885 w							750 m	740 s		
Fluoranthene					828 m			773 s	750 s			
4 FUSED RINGS, 9,10 SUBSTITUTED												
9,10-Dimethylbenz(a)anthracene	910 m	880 w	872 w	842 w		808 s	790 w	756 s	740 s		710 m	680 m
5 FUSED RINGS												
Dibenz(a,h)anthracene		889 s	868 w			818 s		755 s	741 s			668 m
Benzo(a)pyrene		880 m	870 s	850 m	836 s	820 m		755 s	741 s		715 w	688 s
Perylene		890 w				815 s		767 s				
Pentacene	908 s	890 m		842 w	838 m			755 w	741 m	730 s		
3-Methylcholanthrene			870 s		832 m	802 s	790 s	778 w	750 s			685 w

[a] The notations, s, m, w, indicate bands of strong, moderate, and weak intensity respectively.

Frank Hochgesang and Roy Edwards of the Socony Mobil Oil Co., Inc. for furnishing some of the samples for this investigation.

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Submitted March 31, 1961

Errata

The Use of Scatter Diagrams in Emission Spectrography

G. Holdt and A. Strasheim

Vol. 14, 64 (1960)

page 64, Table I and page 69, column 1, formula [7] should read:

$$\text{Worth} = \frac{(s_a)^2 - (s_{st})^2}{2\, s_{a,st}} \pm \sqrt{1 + \left\{ \frac{(s_a)^2 - (s_{st})^2}{2\, s_{a,st}} \right\}^2}$$

Infrared Spectra of Vitamins, A Schiff Base, and an Amino Acid Chelate in Water

Frank S. Parker

Vol. 15, 96 (1960)

page 98. Fig. 8, column 2: Legend should read Cu(l-Lysine)$_2$, 10% solution in 0.027 mm, BaF$_2$ Cell vs. Transmittance Screen

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Notes

A Possible Method of Pulverizing Mica and Other Flaking Materials for Spectrographic Analysis

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Recently a method using the Wig-L-Bug† was described for grinding wool samples to a fine powder for use in the KBr disk technique for infrared spectral study *(1)*. The possibility of using this grinding technique for mica was investigated, as homogeneous pulverisation of mica samples for spectrographic analysis has long been a problem *(2)*. This technique was found to be very satisfactory.

To estimate the efficiency of the new technique, which consists of grinding in a special steel vial (Figure 1) completely filled with carbon tetrachloride, samples ground this way were compared with samples ground dry in the same vial and with samples ground by hand in an ordinary mortar. In the vial 200 mg samples were ground with two steel balls for 30 min. The efficiencies of the three grinding methods are illustrated in Figures 2-4. The photographs were taken with a Zeiss stereomicroscope and were enlarged 300 times with the samples being illuminated by reflection. The procedure which gave good contrast and little conglomeration of the particles in the pictures, was as follows:

A polaroid plate was put on a piece of black felt paper.

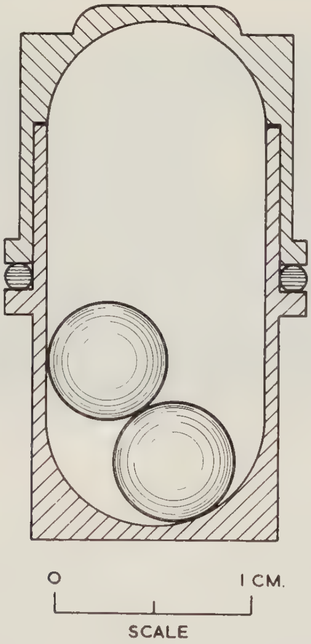

FIG. 1. STEEL VIAL USED FOR LIQUID GRINDING

The sample, immersed in a 50,50 glycerol-water mixture, was placed on the polaroid plate and covered with a microscope glass slide. This sample holder was then illuminated with polarized light at an angle of about 60°. Before taking a photograph, the polarizer in front of the illuminating lamp was adjusted to minimize the amount of unwanted light reflected from the sample holder. Vibrations were avoided by opening the shutter in complete darkness and then switching on the illuminating lamp. The time of exposure was 8 min. This procedure gives a

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†Crescent Dental Manufacturing Co., Chicago, Ill.

FIG. 2. MICA GROUND WET IN VIAL COMPLETELY FILLED WITH LIQUID

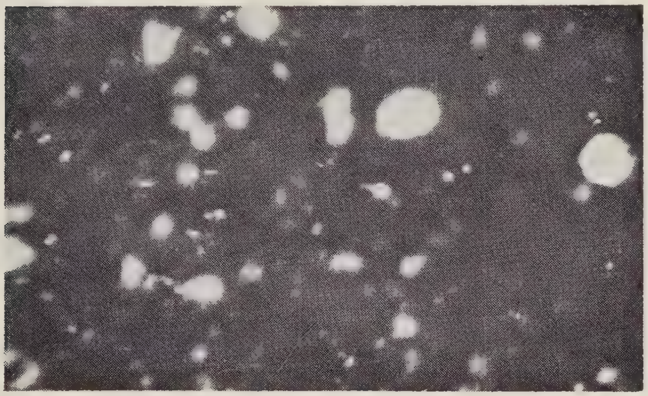

FIG. 3. MICA GROUND DRY

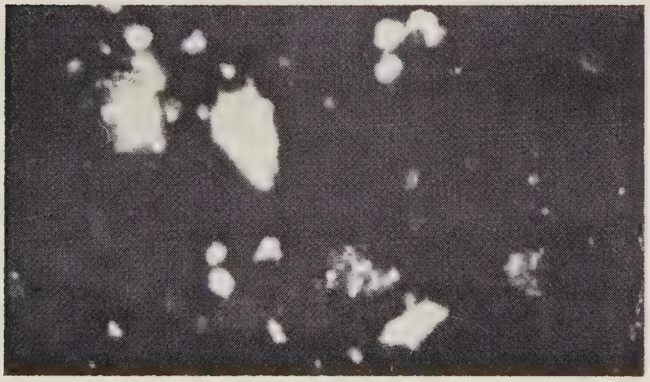

FIG. 4. MICA GROUND BY HAND

photomicrograph of whole particles, while a polarizing microscope would show only the crystallites.

It can be seen from the Figures that two conclusions can be drawn. Firstly, wet-ground samples are more homogeneous than the others, which contain quite a number of large particles. The sieving operation used by Plastinin *(2)* to obtain uniform samples is thus unnecessary. Secondly, iron cannot be determined in samples prepared by this wet-grinding technique. Bright round spots in Figures 3 and 4 probably originate from iron particles. It was also found that the dry-grinding technique produces more contamination from the steel vial than does the wet-grinding technique—samples ground dry were much darker in colour than those ground wet.

Using agate and polystyrene vials with two agate balls, no effective grinding was observed after 30 min. This is probably due to the low specific gravities of these materials. The specific gravities of agate and steel are 2.58 and 7.82 respectively. In this respect considering its high specific gravity (15.63) and hardness, tungten carbide might be very well suited for this type of milling.

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Submitted August 25, 1960

A Limitation Regarding the Use of Ethanol Stabilized Chloroform as a Solvent for Infrared Spectroscopy

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It was observed in this laboratory that the infrared spectra of many compounds, transparent in the region from 2.8 to 3.0 microns in both carbon tetrachloride and carbon disulfide solutions, showed a weak absorption band in this region when their infrared spectra were run as chloroform solutions (Chloroform, Mallinckrodt, Anal. Reag., 0.75% of ethanol added as a preservative), employing the same grade of chloroform in the reference cell. This fact precluded the use of chloroform as an infrared solvent when it was desired to detect small amounts of —OH or > NH in the 2.9 micron region.

When the chloroform used as the solvent was freed from ethanol by washing it with concentrated sulfuric acid (1), the absorption in the range 2.8 - 3.0 microns was no longer observed. An alternate procedure for the removal of small amounts of ethanol from chloroform in-

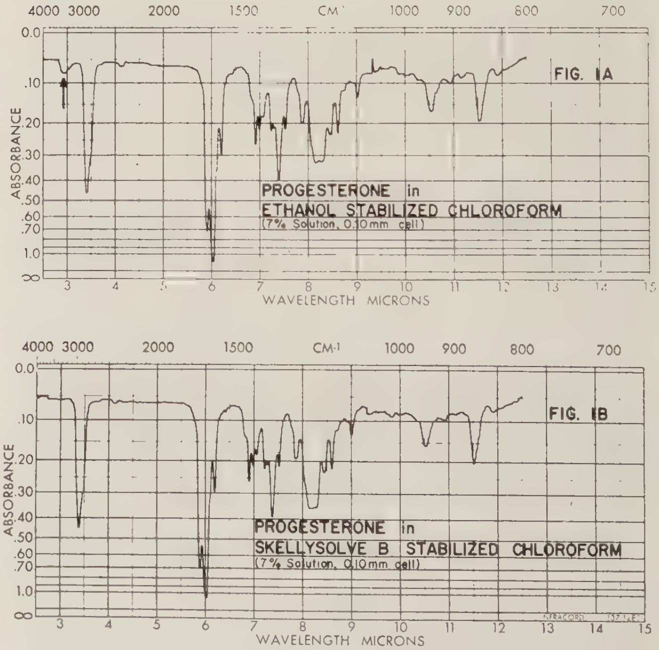

FIG. 1. SPECTRA OF PROGESTERONE IN STABILIZED CHLOROFORM SOLUTIONS

volved the filtration of the analytical reagent chloroform (1100 ml) through a column of alumina (300 g, "Woelm", grade I) *(2)*. It was found that the chloroform freed from ethanol could be stabilized by the addition of 1% (by volume) of Skellysolve B. The chloroform stabilized in this manner could be used as a solvent for a period of at least 8 months with no apparent change in the spectra.

Figure 1 illustrates the above observations in the case of Progesterone, U.S.P. The infrared spectrum was first determined in chloroform, which had been stabilized with 0.75% of ethanol (Figure 1A). A second spectrum was determined in ethanol-free chloroform stabilized with 1% of Skellysolve B (Figure 1B). It is noteworthy that both spectra of the compound are virtually superimposible except for the absorption band at 2.9 microns.

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Submitted October 17, 1960

Spectrochemical Analysis of Oils Using "Vacuum Cup" Electrode

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Zink (*1*) described a new method for handling solution samples in spectrochemical analysis. This method was based upon a suggestion by Eichhoff (*2*) in which he proposed the use of a "capillary" electrode to bring the solution to the analytical gap. The method was modified by changing some of the design features of the capillary electrode and adding a Teflon cup. Attempts to obtain spectra of various oil samples by this method failed; this appeared to be due to the size of the axial canal, and the eventual flaming of the oil sample which seriously disrupted the excitation process.

In order to meet the need for a method to analyze oils spectrographically the "Vacuum Cup" method was modified in the following manner so that it would produce satisfactory spectra. This was accomplished by first redesigning the electrode as shown in Figure 1; note that the axial canal is made with a No. 51 drill and the feed hole is made with a No. 42 drill. With these modifications it was possible to "lift" the oil sample 2¼ in. The second modification was to cut the electrode in such a manner that the Teflon cup would fit on the lower end of the electrode. With this arrangement it was possible to have the Teflon cup and oil sample below the excitation stand as shown in Figure 2. The fire hazard is thus removed.

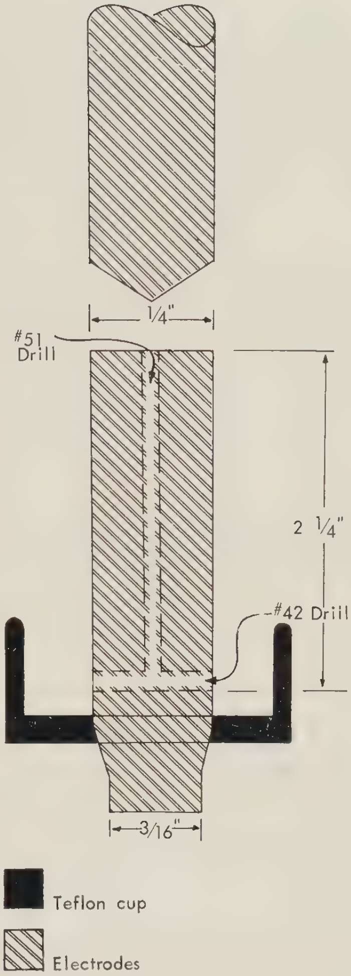

Fig. 1. Modified "Vacuum Cup" Electrode

In these preliminary studies it was possible to detect 0.05% cobalt, 0.23% lead, and 0.07% magnesium in a sample of lubricating oil. The spectrographic conditions and apparatus used are given in Table I.

Table I. Apparatus and Spectrographic Conditions

Analytical gap, mm	5
Excitation unit	ARL Multisource ac Arc
Capacitance, μfd	60
Resistance, ohms	15
Source selector	Low voltage
Discharge point control	90
Power circuit voltage	250
Spectrograph	
Wavelength range, A	2400 to 4130
Slit width, μ	60
Filter	None
Intensity control	10%
Photographic emulsion	Eastman SA-2
Developer	Kodak D-19, 20°C, 3 min

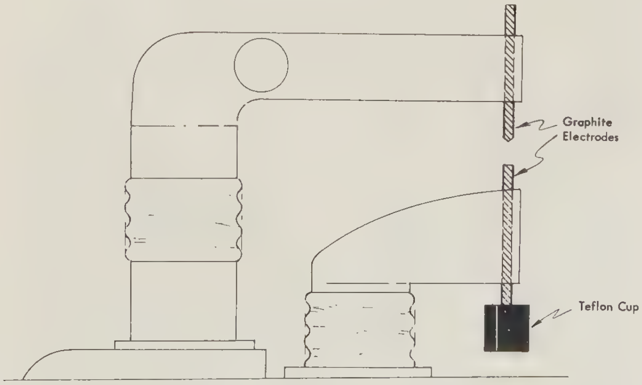

Fig. 2. Arrangement of Electrodes in the Spark Stand

This development will enable the determination of inorganic materials found in oil samples, without going through the ashing process, which is very time consuming and presents many analytical problems. This is a preliminary report of our findings; a more complete report will follow upon further investigations into the application of the "Vacuum Cup" method for the analysis of oil samples.

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Submitted November 23, 1960

Aqua Regia Off-Gases by Infrared Spectrophotometry

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As a part of a program aimed at the recovery of gases given off in the Darex process, a process involving the dissolution of stainless-steel-clad reactor fuel elements in aqua regia (*6,7*), a study was undertaken of the rate of evolution and chemical nature of the gases evolved under high reflux conditions from boiling aqua regia, both with and without metal present. The analytical technique employed infrared and visible spectrophotometry and gas chromatography; it afforded a quantitative measure of the stable gases N_2O, NO, NO_2, NOCl, Cl_2, HCl, HNO_3, N_2, O_2, H_2, and H_2O. Of these, HNO_3 and O_2 were never detected, and one compound existing in very small proportions was unidentified.

Infrared spectra were established for NO, N_2O, NO_2, NOCl, HCl, HNO_3, and water vapor with a Perkin-Elmer Model 21 Infrared Spectrophotometer using sodium chloride optics. Then standard absorption curves for each of the suspected gases were prepared using known concentrations of them. The gas cell had silver chloride windows. HNO_3 gas was obtained by heating $NaNO_3$ with concentrated H_2SO_4. Commercial gases were used in other cases; that they were of acceptable purity was established by comparison of experimental spectra with data in the literature, for example, Pierson, *et al.* (*5*). For quantitative analysis, wavelengths were selected, at an expansion of 16 in./micron, as follows: 2.888 microns for N_2O; 3.288 and 6.119 microns for NO_2 at concentrations greater than 4% and at 4% or less; 5.234 microns for NO; 4.725 microns for NOCl; and 3.556 microns for HCl. A total pressure of 730 mm in the gas cell was employed with nitrogen gas as the diluent.

Since Cl_2 does not absorb in the infrared region, its quantity was measured by the absorption of visible light at 335 mμ using a Beckman Model B Spectrophotometer. To avoid interference by the other gases, off-gases were first exposed to saturated $NaSO_4$. The sulfate decreased the solubility of Cl_2 in water, and any N_2O, NO_2, HCl, or NOCl dissolved or reacted to form water-soluble products. Next, air was admitted until no further red color indicating the oxidation of NO to NO_2 was produced. After the NO_2 had dissolved in the sulfate solution, the remaining gas was bubbled through CCl_4 in an optical cell, which dissolved the Cl_2.

TABLE I. OFF-GASES FROM 5M HNO_3:2M HCL AND 2M HNO_3:5M HCL BEFORE AND AFTER ADDITION OF STAINLESS STEEL

Aqua Regia Concentration (M HNO_3: M HCl)	*Sampling Time*[a]	*Gas Composition, %*								
		H_2O[b]	*Cl_2*	*N_2O*	*NO_2*	*HCl*	*NOCl*	*NO*	*N_2*	*H_2*
		Aqua Regia alone[c]								
2:5	0-1	4.5	67.1	0	0	0	28.4	Trace	0	0
2:5	0-1.5	3.3	33.3	0	0	0	63.4	0	0	0
2:5	0-3	2.8	25.2	0	0	0	72.0	0	0	0
2:5	1.5-10	3.3	5.9	0	0	0	90.8	0	0	0
2:5	0-12	3.0	9.4	0	2.6	0	85.0	0	0	0
2:5	3-18	2.9	6.9	0	0	0	90.2	0	0	0
2:5	15-32	3.0	4.0	0	4.2	0	88.8	0	0	0
2:5	25-44	4.5	7.3	0	0	0	88.2	Trace	0	0
2:5	65-85	2.8	10.8	0	0	0	86.4	0	0	0
5:2	0-1	4.5	53.1	0	0	0	42.4	Trace	0	0
5:2	0-7.5	3.5	40.8	0	0	0	55.7	0	0	0
5:2	0-12	2.9	8.4	0	3.3	0	85.4	0	0	0
5:2	9.5-22.5	3.5	7.5	0	0	0	89.0	0	0	0
5:2	25-30.5	4.5	11.5	0	0	0	84.0	Trace	0	0
5:2	140-234	3.4	15.2	0	0	0	81.4	0	0	0
Aqua Regia with stainless steel (5 g/100 ml) added after the acids had boiled 104 min.										
2:5	113-134	3.0	3.0	24	5.4	0	15.0	57	0	0
5:2	113-134	2.9	4.0	Trace	5.7	0	7.0	72	10	0.5
Aqua Regia with stainless steel (5 g/100 ml) added before heat was applied.										
2:5	20-41	3.0	1.7	7	9	0	37.0	0	40	3.4
5:2	1.4-2	3.0	4.4	2	12.2	2.9	45.6	2	28	0.2
5:2	2.5-4	3.0	2.5	2	16.3	3.6	41.9	4	28	0.3

[a] The interval between initiation of boiling and sample collection, min.
[b] Calculated from vapor pressure of water at the temperature of the collected off-gases.
[c] Composition normalized to 100%.

A Perkin-Elmer Model 154C vapor fractometer with a 2-m molecular sieve column was employed in the quantitative determination of H_2, N_2, and either O_2 or NO (the latter not existing simultaneously to a degree detectible here). Argon was used as the carrier gas. The water-soluble gases were removed by bubbling off-gas samples through a water column.

The instantaneous rate of gas evolution was measured with a calibrated rotameter, and gas samples were collected in collapsed Teflon bags. All measurements and collections were made with a water-cooled reflux condenser between the reaction vessel and the point of collection. This system was preflushed with nitrogen to prevent air-oxidation of NO. Because some of the gases can diffuse through the bags (*1*), analysis was made as rapidly as possible, always within less than one hr. Stainless steel, Type 304, and two aqua regia concentrations (5*M* HNO_3:2*M* HCl and 2*M* HNO_3:5*M* HCl) were tested.

Results are presented in Table I and Figures 1 and 2. As shown, Cl_2 is the major constituent initially evolved, comprising 67% of the 2:5 off-gases during the first min of boiling and 53% of 5:2 off-gases during the same period. NOCl very rapidly replaces it, and in samples collected during the first 12 min of boiling, the chlorine was only 9.4% of 2:5 off-gases and 8.4% of 5:2 off-gases. According to Lunge and Pelet (*2*) the 1:1 ratio of Cl_2 to NOCl to be expected from the primary reaction between HCl and HNO_3 is not realized because of the solubility of NOCl in the liquid phase. The changes in gas evolution rate and in NO particularly are in accord with the stronger oxidizing power of the more concentrated nitric acid, but it is noteworthy that 1.250 g of a 5-g sample of steel remained undissolved by 100 ml of 5:2 aqua regia while

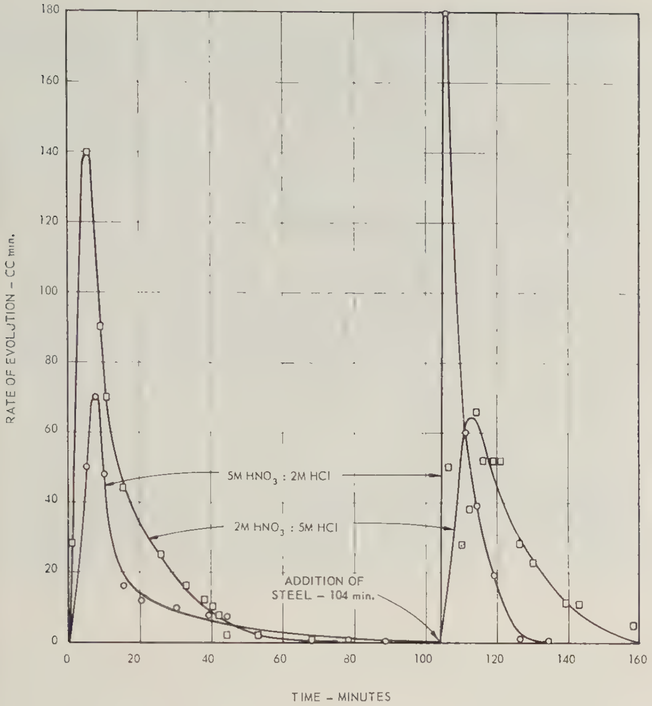

Fig. 1. Rate of Gas Evolution From Boiling Aqua Regia Before and After Addition of Stainless Steel

100 ml of 2:5 aqua regia dissolved 5 g of the steel. Probably the loss of a large portion of the hydrochloric acid as free Cl_2 before the stainless steel was introduced accounts for the difference. The complexing ability of the chloride ion is a major factor in the effectiveness of aqua regia as a solvent, and the available chloride of the 5:2 aqua regia was considerably depleted during the preliminary boiling. In experiments in which the stainless steel was added to aqua regia before heat was applied, all of the steel dissolved in both 2:5 and 5:2 aqua regia.

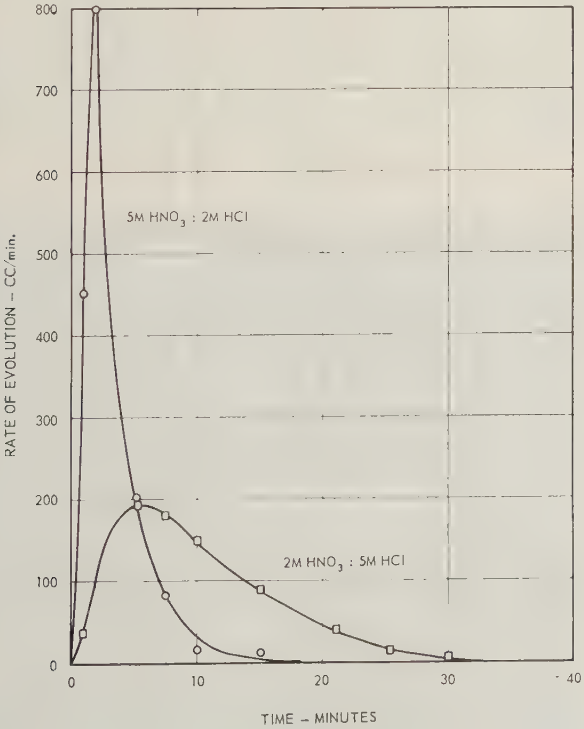

Fig. 2. Rate of Gas Evolution From Boiling Aqua Regia With Stainless Steel Initially Present

An unidentified component appeared as a double peak at 5.91 and 5.96 microns in the infrared spectra of several samples, both before and after the addition of stainless steel to the aqua regia. This peak did not occur in the spectra of HNO_3 and NH_3 obtained in this laboratory nor in the N_2O_5 spectrum reported by Nightingale, *et al.* (*3*).

This investigation was supported by the Oak Ridge National Laboratory operated by Union Carbide Corporation for the U.S. Atomic Energy Commission. More complete information on this work is contained in a report submitted to the sponsor (*4*). While not without its weaknesses, the scheme of analysis as outlined provides a method for following the changing system of off-gases from aqua regia reactions.

The authors wish to acknowledge the helpful suggestions of L. M. Ferris, ORNL, and the assistance of Clyde T. Young who made the spectrophotometric measurements.

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Submitted February 2, 1961

A Polaroid Attachment For A Medium Quartz Spectrograph

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Bryan (*1*) and Bryan and Runge (*2*) have applied the Polaroid emulsion to emission spectroscopy. Although no details of construction are given in these applications, it is apparent that no provision is made for the location of the film in the oblique angle, curved field of the medium quartz spectrograph. Therefore, a commercially available Polaroid Land camera back was modified so that it could be used interchangeably with conventional 4″x10″ spectroscopic plates without any alteration of the optics of such a spectrograph.

The smaller film area of a Polaroid emulsion compared with the 4″x10″ glass plate necessarily decreases the available spectral range to 4¼ in. This, however, is not a serious loss, since it is well recognized that very often approximately half of the 10-in. common spectroscopic plate is wasted when exposed on a medium quartz spectrograph. Experience over a number of years has shown that the spectral range above 3500 A has been of very little value due to the cyanogen band interference and to poor dispersion. The spectral range below 2288 A has very little to offer; first and foremost, because of the anemic character of most spectra in that region and second, because of the paucity of strong, useful, and indispensable lines.

The Polaroid negative as incorporated in this attachment is capable of covering the range 2250 to 3500 A. Within this region will be found the U_1 lines of Cd, Be, Au, B, Sn, Mg, Si, Pt, Bi, Cu, Ag, Ta, and Ni; the U_2 lines of Te, P, Ge, V, Li, and Zn; the U_3 lines of Ga, Al, In, and Na; and the useful Fe 3020-3021 A grouping.

The spectral range can be selected without future choice by permanently positioning the Polaroid back over any area on the original plate holder. This can be ascertained by placing a glass plate with a known spectrum in the normal position in the plate holder and marking off on the curved track 4¼ in. of desirable spectral range. Although the negative spectrum will be 4¼ in., the positive will be masked down so that only 3¾ in. will show. However, there is a choice as to whether to sacrifice one end or the other, i.e., the ½ in. of negative can be sacrificed at either the low (equivalent to 60 A) or high (equivalent to 200 A) wavelength region. This choice is made at the moment of development and can be changed with each frame.

The term "frame" is used as being synonymous with the term "exposure" in the photographic arts.

The curved plate track of the Hilger plate holder must be relocated so as to give proper (3⅝ in.) track width to accommodate the Polaroid negative. Based on the marking when the spectral range is chosen, metal rollers R_1 and R_2 are placed as shown in Figure 1. These rollers are spring-loaded so as to maintain the negative snugly on the track. Spring fingers are added to each track to aid in holding the negative taut on the curved field. (The slide has been removed for constructional convenience.) Figure 1 also shows aluminum angle cleats upon which the Polaroid back will be fastened.

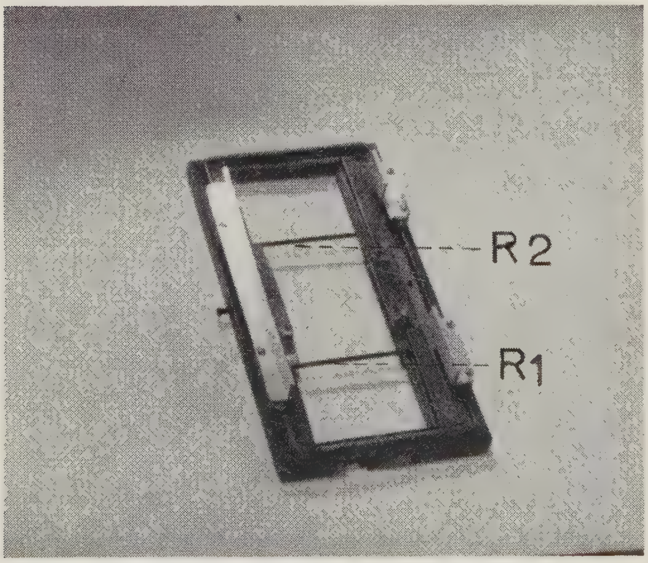

FIG. 1. PLATE HOLDER BEFORE ATTACHING CAMERA
R_1—Roller 1, R_2—Roller 2

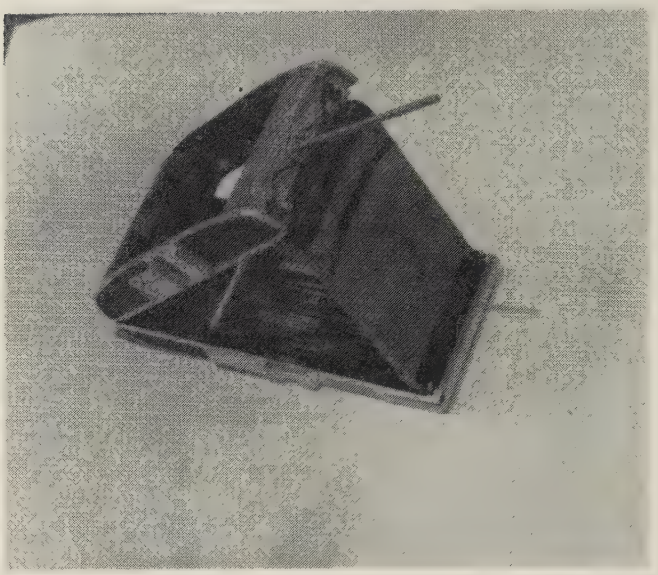

FIG. 2. CAMERA WITH LEVER LATCH MODIFICATION

The Polaroid Land camera back (front cover, bellows, and braces removed) is modified as follows: a slot S_1 is cut at the negative end so as to allow the negative to move through freely from the original negative well to roller R_1 as shown in Figure 3. At the positive side, a slot S_2 is cut parallel to S_1 at the point indicated but not visible in Figure 3. A roller R_3 is located on the medial side of, adjacent and parallel and in the same large plane as S_2.

The lever latch is modified by cutting off the thumb end so as to leave a metal circle about ¾ in. diam. As shown in Figure 2, a 3/16-in. diam. shaft is fastened to the circle so that rotation can be effected by shaft torque instead of the finger lever. Finally, the shaft is bent and further braced as shown in Figure 3.

The Polaroid back is fastened to the plate holder so located that the negative slot S_1 is over roller R_1 while slot S_2 is located over roller R_2. Small aluminum plates are attached in the open spaces between plate holder and Polaroid back. Light leaks are patched with epoxy cement.

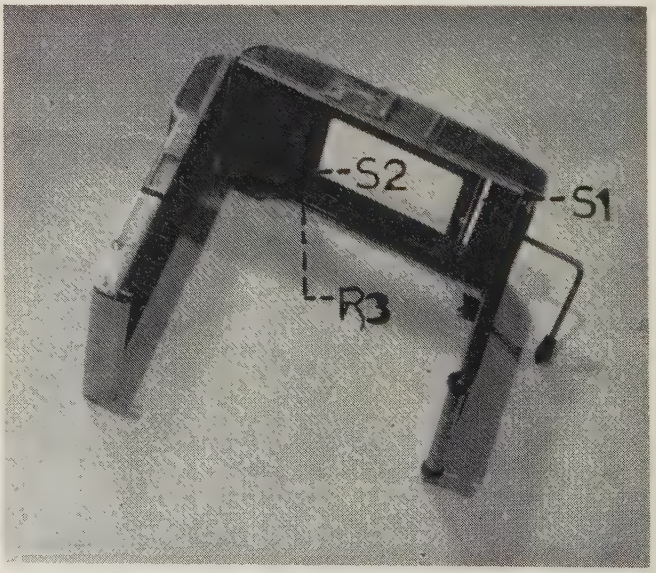

FIG. 3. CAMERA MODIFIED WITH SLOTS AND ROLLERS
S_1—Slot 1, S_2—Slot 2, R_3—Roller 3

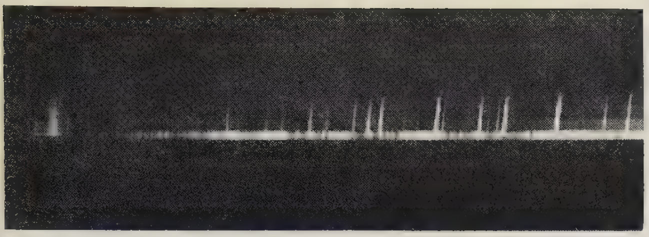

Fig. 4. Cadmium Spectrum Favoring Low Wavelength

Before placing the roll on the camera, the end (leader) of the black protective paper of the negative roll is sharpened by cutting off triangles about 1½ in. from each corner. The negative roll is separated from the positive roll, the former is dropped into its receptacle (well) and the sharpened end threaded through the slot S_1 down between the track and roller R_1, along the track and over roller R_2, up over the medial side of roller R_3, through slot S_2, and finally over the conventional Polaroid roller. The negative is pulled further until the original point of positive attachment to the negative is reached. The negative is backed up one in. at which point the positive is fastened by means of pressure sensitive tape. The system is now treated as described in the original Polaroid directions accompanying the camera.

Following exposures made in the usual way, the cutting bar is lifted, exposing the ends of the positive and negative. The negative only is pulled out to extend from 1 to 1¾ in. beyond the positive. (The smaller will favor the low wavelength region: the larger will show more of the long wavelength.) The positive release button is pushed, and both positive and negative are pulled out together.

Occasionally the positive and negative rolls are practically cemented together by the development jelly of the previous frame; hence the negative cannot be pulled separately from the positive. This difficulty can often be overcome by inserting a thin stainless steel spatula between negative film and positive paper frame through the open picture door. A better solution is to discard one frame, if inspection through the picture door discloses a hardened, glue-like effect of the developing jelly that is a condition resulting from a long unused period.

Seven frames can be obtained from one 8 "exposure" roll. One frame is sacrificed to compensate for the greater travel path of the negative.

Figure 4 shows the cadmium spectrum which includes highly self-absorbing Cd 2288 A. Also shown at extreme left are Cd 2267 A and Cd 2265 A. This extent into the lower wavelength region compares favorably with common spectroscopic plates. Figure 5 shows the copper spectrum from a frame slanted towards the higher region with the prominent CN band head at 3590 A. A Hartmann diaphragm iron spectrum is included in each exposure.

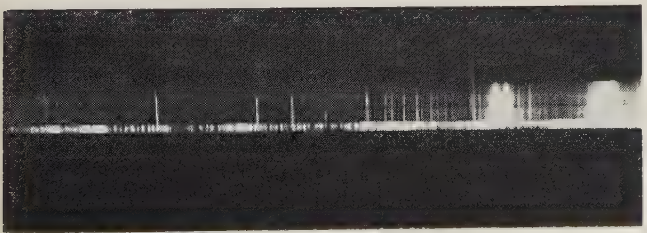

Fig. 5. Copper Spectrum Favoring High Wavelength

Literature Cited

(1) F. R. Bryan, J. Chem. Ed. **37**, 471 (1960)

(2) F. R. Bryan and E. F. Runge, Applied Spectroscopy **15**, 16 (1961)

Submitted February 15, 1961

Spectroscopic Tricks

Modification of the Coleman Model 14 Universal Spectrophotometer to Simplify Wavelength Calibration Adjustment

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The difficulties encountered in recalibrating the Coleman instrument, either after replacing the exciter lamp or if the wave length has shifted, are familiar to most users. These difficulties may be overcome by a few simple modifications.

1. An outside control for adjusting the wave length calibration while the instrument is in its normal position is installed as follows: The calibration screw G (as shown in the Figure 1) and retaining wire under *F*, that ordinarily meshes with the knurling on *G*, are removed. A new screw with a socket head (Figure 2, all materials are brass) is soldered to a 13-14 in. long flexible shaft, which may be a ⅛ in. speedometer cable obtainable from any auto supply store. The new calibration screw is then threaded into the place previously occupied by *G*. To prevent whip, the flexible shaft is run through a plastic clip fastened by an ⅛ in. long sheet metal screw at position *S2*. Before attaching the adjustment knob to the flexible shaft, a spring tension washer ⅝ in. diam., 7/16 in. hole, 0.012 in. thick is slipped over the shoulder, the knob is then inserted into the hole, and a collar ⅝ in. diam. 11/32 in. hole, ⅛ in. thick is put on, followed by a 5/16-18 NC nut. The outer end of the flexible shaft is fastened by means of 2 set screws to a similar but unthreaded adapter that is part of the adjustment knob. The

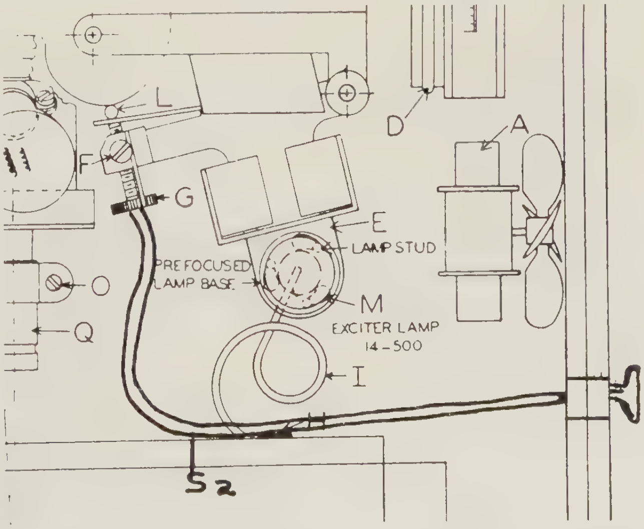

Fig. 1. Part of Interior of Model 14 Universal Spectrophotometer

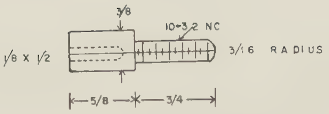

FIG. 2. FLEXIBLE CABLE ADAPTER

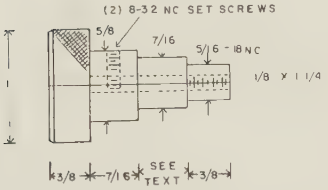

FIG. 3. ADJUSTMENT KNOB

length of the shoulder of the knob is equal to the wall thickness plus the spring tension washer plus 0.005 in. (Figure 3). This shoulder rides in a hole of the proper size in the wall of the case. The hole is drilled about 3⅝ in. from the bottom of the case and 3¼ in. from the back; in other words, half-way between the O and BLK knobs and 1¾ in. up, measured from the centers. Care must be taken that there is no interference with the movement of the cooling fans. Trial and error will soon show the degree of tension required.

2. The battery is fastened to the case by a brass strap 1 in. wide, 7¼ in. long, 0.032 in. thick, bent to form a channel cross-section whose arms are 2⅜ in. and the base is 2½ in. The ends of the strap are soldered into the slotted heads of two 6/32 NC brass screws. A pair of No. 28 holes on 2⅝ in. vertical centers are drilled through the case, the lower one is 6¾ in. in from the front of the case and 1¼ in. from the bottom.

The author wishes to express his appreciation to William F. Hafner of this Institute for his coperation and mechanical skill in making the necessary attachment and to Coleman Instruments Inc. for permission to reproduce part of Figure 1.

Submitted November 30, 1960

Infrared Quantitative Analytical Data

Publication is open to any person and is not limited to members of the Coblentz Society or the Society for Applied Spectroscopy. Contributions should be sent directly to the Chairman of the Review Committee, Dr. A. Lee Smith, Spectroscopy Laboratory, Dow Corning Corporation, Midland, Michigan. It is requested that methods be submitted in quintuplicate, using the standard format. Infrared spectra cannot be published as a part of this program.

Determination of Sodium Salts of C_1-C_3 Carboxylic Acids

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CS-162

Component			Range %	Accuracy %	λ or ν B.L. Pts.	Slit (mm) Δλ or Δν	Conc. Length
No.	Name	Formula					
1	Sodium Acrylate	$C_3H_3O_2Na$	0-100	±2	7.80μ	0.103 0.030μ	0.5 wt.% 1 mm
2	Sodium Acetate	$C_2H_3O_2Na$	,,	,,	10.83μ	0.184 0.038μ	,,
3	Sodium Formate	$C_1H_1O_2Na$	,,	,,	12.27μ	0.245 0.047μ	,,
4	Sodium Propionate	$C_3H_5O_2Na$	,,	,,	12.92μ	0.285 0.053μ	,,

Instrument: Perkin-Elmer Model 21, NaCl Prism
Sample Phase: KBr Disc
Cell Windows:
Absorbance Measurement: Base Line......X...... Point...........
Calculation: Inverted Matrix........... Successive Approx............
Graphical......X......
Relative Absorbances—Analytical Matrix:

Component/λ	7.80μ	10.83μ	12.27μ	12.92μ
1	0.250	0	0	0
2	0	0.184	0	0
3	0	0	0.357	0
4	0.033	0	0	1.433

Material Purity: 99+%

Comments: Base line is drawn from transmittance peak to peak on either side of band. The method is especially useful for quantitative analysis of these acids in low concentration in water solution. A standard titration with sodium hydroxide gives simultaneously the total acid concentration and conversion to the sodium salt. The salt is dried thoroughly, mixed before sampling, and the quantitative split on individual acids obtained as described herein.

Determination of Phenol and p-Chloroaniline in Mixtures of n-Hexane and Methylcyclopentane

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CS-163

Component			Range %	Accuracy %	λ or ν B.L. Pts.	Slit (mm) Δλ or Δν	Conc.% Length (mm)
No.	Name	Formula					
1	Methylcyclopentane	C_6H_{12}	0-50	±2.0	10.30μ	0.121 0.027	10 0.041
2	p-Chloroaniline	C_6H_6NCl	0-25	±2.0	12.27μ	0.192 0.038	,,
3	Phenol	C_6H_6O	0-20	±2.0	13.38μ	0.249 0.047	,,
4	n-Hexane	C_6H_{14}	0-60	±2.0	13.84μ	0.280 0.052	,,

Instrument: Perkin-Elmer Model 21, NaCl Prism
Sample Phase: Solution in Carbon Disulfide
Cell Windows: NaCl
Absorbance Measurement: Base line........... Point..... X......
Calculation: Inverted Matrix........... Successive Approx......X......
Graphical.. . . .

Relative Absorbances—Analytical Matrix:

Component/λ	10.30μ	12.27μ	13.38μ	13.84μ
1	0.208	0.035	0.008	0.001
2	0.180	7.280	0.500	0.420
3	0.320	1.350	12.950	0.630
4	0.035	0.034	0.045	0.330

Material Purity: Methylcyclopentane and n-Hexane - Phillips Pure Grade 99 mole % minimum; Phenol - Monsanto 99%; p-Chloroaniline - Eastman 98%

Comments: Relative absorbances are given as the slope of the Beer's Law concentration curves used and are expressed in terms of absorbances per 100% of constituent.

Society News

The Governing Board at the meeting held in New York City on November 15, 1961 approved at the first reading a number of amendments and additions to the Society's Constitution and By-Laws.

Several of these changes (Articles II and III of Constitution) are required so that the Society can qualify as a tax exempt scientific society under the terms of the Internal Revenue Act (1954). The other amendments and additions are intended to bring about a more effective and efficient operation of the Society's business and program.

The membership will shortly have an opportunity to vote for the adoption or rejection of the proposed changes.

Article II—Object

As Now Written	*Recommended Amendments and Additions to the Constitution*
The objective of this organization shall be: (a) To advance and disseminate the knowledge and information concerning the art and science of spectroscopy, and other allied sciences. (b) To advance the professional standing and growth of the Society and its members. (c) To coordinate cooperative endeavors of its individual members and sections. (d) To promote and maintain a close bond among its members.	Change to read The objective of this organization shall be: To advance and disseminate knowledge and information concerning the art and science of spectroscopy and other allied sciences, conforming to the provisions of Section 501 (c) (3) of the Internal Revenue Code (1954).

Article III—Scope

The scope of this Society shall be to undertake and promote activities which shall benefit the membership and the Local Sections. The term Spectroscopy as used in the title and body of this Constitution is understood to mean the science and art of absorption, emission, Raman, mass and related forms of spectral study for determining the composition and structure of matter.	Change to read The scope of this Society shall be to undertake and promote activities which shall accomplish the objective. The term spectroscopy as used in the title and body of this Constitution is understood to mean the science and art of absorption, emission, Raman, mass and related forms of spectral study for determining the composition and structure of matter. To accomplish the objective stated in Article II, The Society and/or Local Sections may conduct conferences or symposia on scientific subjects and may, in addition to the Journal as provided in this Constitution, publish or give financial support to the publication of other scientifically useful information pertaining to spectroscopy.

Article V—Local Sections

Section 3—The Constitution and By-Laws of such local Sections shall be consistent and in harmony with the Constitution and By-Laws of the Society.	Change to read Section 3—The Constitution and By-Laws of such local sections shall be consistent and in harmony with the Objective and the Constitution and By-Laws of the Society.

Article VI—Government

Section 6—The term of office for the elected officers shall be as follows: The President one year, the President-Elect one year, the Secretary three years and the Treasurer three years. The elected officers shall begin their respective terms of office at the beginning of the fiscal year but will be installed at the Annual Meeting.	Change to read Section 6—The term of office for the elected officers shall be as follows: The President one year, the President-Elect one year, the Secretary three years and the Treasurer three years. The terms of the Secretary and Treasurer shall whenever possible commence on different years. The elected officers shall begin their respective terms at the beginning of the fiscal year but be installed at the Annual Meeting.
	Add Section 7—The elected officers and the immediate Past President shall constitute an Executive Committee which shall assume responsibility for the government and welfare of the Society in the interim between the Governing Board meetings.
Section 7—Vacancies in the elective offices, except that of President, shall be filled until the next Annual Meeting by a letter ballot election conducted by the Secretary, who shall submit to the members of the Governing Board the names of candidates selected by the Nominating Committee. A majority of the votes cast shall be required for election.	Change to read Section 8—Vacanies in the elective offices, except that of President, shall be filled until the next fiscal year by a letter ballot election conducted by the Secretary, who shall submit to the members of the Governing Board the names of candidates selected by the Nominating Committee. A majority of the votes cast shall be required for election.

Article VII—Duties of the Officers

Section 3—The duties of the Secretary shall be to maintain the approved records of the Society and a correct register of the members of the Society. He shall attend to the correspondence of the Society and issue notices of all meetings as set forth by the President and Governing Board. He shall conduct the letter ballots under the direction of the Governing Board and shall submit the Ballots to the Tellers appointed by the President. The Secretary shall submit a written report at the Annual Meeting. The Secretary must present bond similar to the Treasurer.	Section 3—delete last sentence.

Article VII—Duties of Officers (continued)

As Now Written	*Recommended Amendments and Additions to the Constitution*
Section 4—The Treasurer shall receive all accounts payable and shall sign and issue all disbursements payable for the Society. The Treasurer must be bonded to an excess of ten percent above the estimated annual operating budget. The Treasurer shall submit a written report at the Annual Meeting which shall be a certified audit.	Change to read Sction 4—The Treasurer shall receive all accounts payable and shall sign and issue all disbursements payable for the Society. The Treasurer shall submit a written report at the Annual Meeting. Add Section 5—The fiduciary officers of the Society and the Society's Journal and/or other ventures involving the collection or distribution of money shall each be bonded to the extent of not less than twenty per cent of the estimated total annual amounts of their accounts. The cost of the said bonding shall be paid by the Society. present Section 5 to renumbered Section 6.

Article VIII—Dues

Section 3—All Dues are payable by the end of the fiscal year.	Change to read Section 3—All dues are due and payable on or before the beginning of each fiscal year. Provisions for the suspension for Non Payment of Dues shall be set forth in the By-Laws.

Article X—Meetings

Section 2—The Governing Board shall convene at least twice a year in addition to the Annual Meeting at times and places designated by the President. A four week's written notice of these meetings shall be given to all members of the Governing Board.	Section 2—Change to read "at least once a year" instead of "at least twice a year."

Article XII—The Journal

Section 3—The Journal shall operate on a budget prepared by the Editor-in-Chief and Business Manager and approved by the Governing Board. The finances of the Journal shall be handled in the manner set forth in the By-Laws. An annual report of this account shall be prepared and submitted to the Treasurer and Auditing Committee.	Section 3—Delete last sentence.

Article XIII—General Provisions

	Add Section 3—The accounts and records of the Secretary, Treasurer, Business Manager of the Journal and any other financial transactions in the name of the Society shall be subject to audit by a Certified Public Accountant approved by the Committee on Audit, if the Committee deems such an audit necessary.
	Add **Article XVI—Dissolution** In event of either voluntary or involuntary dissolution of the Society, the funds or assets of the Society, remaining after discharging all just debts of the Secretary or its officers in the name of the Society, shall be distributed without encumbrances to a non-profit group, organization or institution of learning engaged in the field of spectroscopy within the contemplation of Section 170 (c) (2) of the Internal Revenue Code (1954). The selection of the recipient or recipients shall be made by the majority vote of the Governing Board in office at the time of dissolution but in no event shall the assets be distributed to any member or members of the Society.
Article XVI—Amendments	Change to **Article XVII—Amendments**

Article II—Duties of the Officers

As Now Written	*Recommended Amendments and Additions to the By-Laws*
Section 3—Secretary	Change to read
(e) He shall receive all monies, keep an accurate account thereof, and pay the same promptly to the Treasurer, taking his receipt therefor.	(e) He shall submit a bill for the annual dues and issue a membership card to each member at least two months prior to the beginning of the next fiscal year.
(f) He shall bill all members for dues and issue a membership card on receipt of payment thereof.	(f) He shall receive all monies for the payment of dues, keep an accurate account thereof and promptly deposit the same to the Society's general fund at the same time transmit a copy of the records of the transaction to the Treasurer.

Article II—Duties of Officers (continued)

As Now Written	*Recommended Amendments and Additions to the By-Laws*
(h) He shall supply each Local Section with a roster of its membership after January 2nd of each year.	(h) He shall monthly transmit a roster of the members in good standing to (1) the Business Manager of the Journal to maintain the circulation lists current, (2) each Local Section the names of the members designated to that Section. He shall at the same times authorize the Treasurer to transfer to the Local Sections that portion of the membership dues allotted to the Local Sections in Article V of these By-Laws.
(i) He shall notify the Treasurer the proper distribution of Sustaining membership dues.	(i) add "not later than March 31st of each year."
(j) He shall order the Treasurer to pay membership dues to the Local Section on March 1, July 1, and December 1st of each year.	delete part (j).
(k) He shall be empowered to hire the personnel to assist in the clerical routines of this office subject to the approval of the Governing Board.	change, present part (k) to become part (j).
(l) He shall, before entering the duties of the office, give a good and sufficient bond (ten percent excess of the Annual Budget). This bond shall be sent to the President no later than two weeks after the Annual Meeting.	delete part (l).
Section 4—Treasurer	Change to read
(a) The Treasurer shall receive all monies from the Secretary, giving him a receipt therefor, and pay all orders drawn him by the officers and Governing Board when properly attested.	(a) The Treasurer shall receive all monies payable to the Society other than dues, giving a receipt or acknowledgment therefor and cause the same to be deposited in the Society's general fund. He shall transfer to the Journal account that portion of the membership dues allotted to the Journal in Article V of these By-Laws. He shall pay orders on the Treasury only upon receipt of a properly attested voucher.
(d) He shall distribute the dues to the Local Sections after authorization by the Secretary.	(d) add "by May 1st and October 1st of each year."
(e) He shall keep accurate accounts of all receipts and expenditures and submit an annual report to the Auditing Committee one month prior to the Annual Meeting.	Change to read (e) He shall keep accurate records of all receipts and expenditures and shall at the Annual Meeting make a summary report and file a complete report of the transactions of the preceding fiscal year.
(f) He shall, before entering upon the duties of the office, give a good and sufficient bond (ten percent in excess of the Annual Budget). This bond shall be sent to the President no later than two weeks after the Annual Meeting.	delete present part (f). Add (f) He shall be empowered to hire the personnel to assist in the clerical routines of this office subject to the approval of the Governing Board.

Article IV—Dues

Section 2—The annual dues of all members are due and payable to the Secretary of the Society on or before March 1st of each year, unless written notice of intent to resign is submitted to the Treasurer before February 1st.	Change to read Section 2—The annual dues of all members are due and payable on or before January 1st of each year unless written notices of intent to resign has been submitted to the Secretary as set forth in Article I of these By-Laws.
Section 3—A member who fails to pay the annual dues on or before May Thirty-first shall automatically be suspended. The member's subscription to the Journal shall be cancelled.	Section 3—change to read "on or before April 20th" instead of "on or before May 31st."
Section 5—A reinstated member shall receive back issues of the Journal for the dues paid only if these issues are available from stock on hand at the time of reinstatement.	delete present Section 5.
Section 6—A Local Section may assess additional dues at the discretion of its Executive Board or as limited by the Section's By-Laws.	renumber as Section 5.

Article V—Allocation of Dues

Section 4—The dues of Supporting Members shall be established and collected by the Local Section.	
(a) Three dollars of each Supporting Member's dues shall be forwarded to the Society's Secretary. Of the amount, one dollar shall be deposited in the general account and two dollars in the Journal account.	Change to read (a) Five dollars of each Supporting Member's dues shall be forwarded to the Society's Secretary. Of the amount, one dollar shall be deposited in the general account and four dollars in the Journal account.

Article VI—Disbursement of Funds

Section 1—No officer or member of the Governing Board shall incur any financial obligations in the name of the Society without the consent of the Governing Board. Regular expenses of the Society shall be paid by the Treasurer, subject to the approval Governing Board.	Change to read Section 1—Regular budgeted expenses of the Society shall be paid by the Treasurer upon receipt of a proper voucher. No officer or member of the Society shall incur any financial obligations in the name of the Society without the consent of the Governing Board or in the interim between meetings the Executive Committee.
Section 3—Expenses of the Society, not specifically spelled out and provided for in the budget, shall be approved by the Governing Board before payment.	Change to read Section 3—Expenses of the Society, not specifically spelled out and provided for in the budget must be approved before payment by the Governing Board or in the interim between meetings the Executive Committee.

Article VII—The Journal

As Now Written	*Recommended Amendments and Additions to the By-Laws*
Section 2—The Business Manager	Change to read Section 2—The Business Manager shall be responsible for maintaining the Journal operation in a sound financial condition. (a) He shall have the prerogative of appointing a staff to assist him.
(a) He shall be responsible for the maintenance of an accurate mailing list of all subscribers.	(b) present part (a).
(b) He shall fix advertising rates subject to the approval of the Governing Board.	(c) present part (b).
	Add (d) The Journal monies shall be deposited in a reliable bank under credit of the Applied Spectroscopy Journal Account; all expenses and bills for operation of the Journal shall be paid by checks drawn by the Journal Treasurer on receipt of an approved voucher or bill.
(c) He shall be empowered to hire the personnel necessary to publish and mail the Journal subject to the approval of the Governing Board.	Change to read (e) He shall be empowered to enter into contracts for the printing and mailing of the Journal, subject to the approval of the Governing Board.
(d) He shall collect accounts receivable, transfer or deposit all monies received to the Society Treasury, issue warrants to the Treasurer authorizing payments from the Journal account.	Delete.
(e) He shall keep accurate records of all transactions and submit the same for audit.	Delete.
	Add (f) He shall prepare a full and accurate report of the business and finances of the Journal and shall if directed submit his records for audit.
(f) He shall, together with the Editor-in-Chief and Budget Committee, prepare a budget for the operation of the Journal subject to approval of the Governing Board.	Renumber as to (g).
	Add Section 10—The Publications Committee shall serve in an advisory capacity to the Editor-in-Chief and Business Manager of the Journal.

Article VIII—Elections

As Now Written	*Recommended Amendments and Additions to the By-Laws*
	Add Section 1—The election of officers of the Society shall be conducted in the manner prescribed in these By-Laws during the month of October. A lapse of not less than twenty one (21) days shall be permitted between mailing out the ballots and the closure of the election. Renumber present Section 1 as Section 2 present Section 2 as Section 3 present Section 3 as Section 4 present Section 4 as Section 5

Article X—Committees

As Now Written	*Recommended Amendments and Additions to the By-Laws*
Section 1—The Nominating Committee	Change to read
(b) The Nominating Committee shall submit the names of the nominees to the Secretary three months prior to the Annual Meeting.	(b) The Nominating Committee shall submit the names of the nominees to the Secretary not later than June 15th of each year.
(c) Additional nominations for any elective office of the Society may be made by a petition signed by at least forty regular members and received by the Secretary not less than sixty days before the Annual Meeting. Nominees by petition shall meet the other qualifications of nominees as stated in the Constitution.	(c) Additional nominations for any elective office of the Society may be made by a petition signed by at least forty regular members and received by the Secretary not later than July 15th. Nominees by petition shall meet the other qualifications of nominees as stated in the Constitution.
Section 2—The Auditing Committee	Change to read
(c) This committee shall be responsible for the audit of all the Society finances.	(c) This committee shall be responsible for the audit of all the Society finances. The committee may approve a Certified Public Accountant to audit the accounts and records of the Secretary, Treasurer, Business Manager of the Journal and other financial transactions of the Society which it deems necessary and advisable.
Section 3—The Budget Committee	Change to read
This committee shall be comprised of one delegate appointed by each Local Section. The President shall designate the Chairman. This committee shall present the budget for each fiscal year to the Governing Board for approval at the Annual Meeting.	This committee shall be composed of seven delegates representing different local sections. The committee shall prepare a budget for the operation of the Society and together with the Editor-in-Chief and Business Manager of the Journal prepare a budget for the operation of the Journal. These budgets shall be presented for the approval of the Governing Board at the Annual Meeting.

Article X—Committees (continued)

As Now Written	*Recommended Amendments and Additions to the By-Laws*
Section 5—The Committee of Tellers This committee shall be comprised of two regular members appointed by the President. This committee shall tally the votes cast in the election of officers, on any proposed amendment to the Constitution or By-Laws, or any resolution voted upon by the regular members and shall report the results to the Secretary of the Society.	Change to read This committee shall be comprised of three regular members appointed by the President. This committee shall tally the votes cast in the election of officers, on any proposed amendment to the Constitution or By-Laws, or any resolution voted upon by the regular members and shall report the results to the Secretary of the Society.
Section 8—The Program Committee This committee shall consist of three delegates appointed by the President. Its duties shall consist in acquiring the programs of the meetings of Local Sections; in compiling a list of available speakers for Local Section meetings, and in assisting the program chairman of Local Sections as requested.	
This Committee shall investigate and coordinate a yearly speaking tour to the Local Sections by an exceptional speaker with a stimulating subject. It shall coordinate the programs of the National Society with or in conjunction with conferences, and symposium with annual meetings. The Chairman shall make an annual report to the Governing Board.	Delete last two sentences of 2nd paragraph.
	Add Section 9—Publications Committee This committee shall be composed of the President-Elect as chairman and six members appointed by the President and approved by the Governing Board. This committee shall serve in an advisory capacity to the Editor-in-Chief and Business Manager of the Journal also any other publications undertaken by the Society. The committee shall present a report of its activities and recommendations to the Governing Board at the Annual Meeting or upon request of the Board.
	Section 10—Conference Committee This committee shall be composed of seven members appointed by the President. This Committee shall review all requests by Local Sections or groups of Local Sections to host a Society sponsored meeting or conference. It shall advise and assist the host group in matters relating to program and arrangements to assure the compliance with the established policy governing meetings sponsored by the Society.
	Add Section 1—The chairman of every standing committee shall be a Local Section delegate at the time of appointment and shall continue as chairman throughout the fiscal year for which the appointment was made even though his or her term as Local Section delegate may have expired. [*if the proposed Section 1 of Article X is adopted—1—the word delegate or delegates which appear in the other Sections of Article X will be changed to member or members—2—The section numbers will be changed to accommodate the new Section 1.*]

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* * *

S.A.S. Local Section News

Editor—Carl J. Leistner

[Items for this column should be sent to the Editor, Mr. Carl J. Leistner, United Carbon Products Company, P. O. Box 747, Bay City, Michigan.]

Pittsburgh Section, S.A.S., and Pittsburgh Analytical Group, A.C.S.

The Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy

Exposition of Modern Laboratory Equipment

	A. M.	P. M.
Monday March 5	Analytical Chemistry—Electrochemical Methods Gas Chromatography—Instrumentation Nuclear Magnetic Resonance	Analytical Chemistry—Automated Methods Symposium on Gas Chromatography—Theory and Capillary Columns Analytical Chemistry—General
Tuesday March 6	Analytical Chemistry—Organic Analysis Gas Chromatography—Analysis of Gases Nucleonics	Symposium on Chemical Analysis of Metals Gas Chromatography—General
Wednesday March 7	Analytical Chemistry—Non-Ferrous Metals. Symposium Commerorating the Fifteenth Anniversary of the Fisher Award in Analytical Chemistry—Analytical Chemistry Clinic Gas Chromatography—General	X-Ray Spectroscopy SSP Award Address—Symposium on Vacuum Ultraviolet Spectroscopy Symposium on the Spectroscopic Study of Polymers
Thursday March 8	Molecular Spectroscopy—Instrumentation Emission Spectroscopy—Instrumentation Symposium on the Spectroscopic Study of Polymers	Emission Spectroscopy—Atomic Absorption, Flame and Gas Analysis Molecular Spectroscopy—General Symposium on the Spectroscopic Study of Polymers
Friday March 9	Emission Spectroscopy—Controlled Atmospheres and Trace Analysis Infrared Spectroscopy—General	Emission Spectroscopy—Standards, Analysis of Metals and Solutions Infrared Spectroscopy—General

Monday Morning—8:30 A. M.

ANALYTICAL CHEMISTRY—ELECTROCHEMICAL METHODS

J. P. McKaveney, Presiding

Coulometric Power Supplies: Some New Approaches—W. T. Barnes and D. E. Wilson, General Dynamics Corp.

Galvanic Determination of Hydrogen—P. Hersch, Beckman Instruments, Inc.

An Improved Electrolytic Hygrometer Cell—J. W. Reeds and A. J. Petersen, Beckman Instruments, Inc.

A Study of Reference Electrode Liquid Junction Potentials—G. Matsuyama, Beckman Instruments, Inc.

Use of the Amperometric Oxygen Electrode for Determining Dissolved Oxygen—R. Jones and H. Watanabe, Beckman Instruments, Inc.

Effects of Slow Chemical Processes in Automatic Redox Titrations—R. E. Cover and L. Meites, Polytechnic Institute of Brooklyn

Differential Potentiometric Determination of Selenium and Tellurium in Refined Selenium Products—S. Barabas and P. W. Bennett, Canadian Copper Refiners, Ltd.

Differential Potentiometric Determination of Tellurium in Refined Tellurium—P. W. Bennett and S. Barabas, Canadian Copper Refiners, Ltd.

Determination of Gold by Controlled Potential Coulometry—L. E. Duncan, General Electric Co.

GAS CHROMATOGRAPHY—INSTRUMENTATION

R. D. Hinkel, Presiding

The Practical Aspects of Column Design—W. J. Curren and E. A. McIntyre, The Foxboro Co.

A Unitized Gas Chromatograph for Repetitive Analyses—R. A. Potter, E. I. Du Pont De Nemours and Company, Inc.

Improvement in Microcoulometric Gas Chromatography—D. M. Coulson, L. A. Cavanagh, E. M. McCarthy, L. J. Salas, and V. B. Wilton, Stanford Research Institute

Dual Column Chromatography Utilizing Ionization Detection—R. D. Condon, The Perkin-Elmer Corp.

A Negative Ion Gas Analysis Technique—V. N. Smith and J. Merritt, Shell Development Co.

The Application of the Electron Capture Detector to the Analysis of Lead Alkyls in Gasoline—R. E. Laramy, L. D. Lively and G. Perkins, Jr., Continental Oil Co.

A Gas Chromatograph Detector Selective for Halogen and Sulfur Compounds—J. C. Sternberg, D. T. L. Jones and R. M. Morris, Beckman Instruments, Inc.

(Continued on page 21A)

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An Electronic Integrator-Digitizer for Gas Chromatography—C. E. Davis and W. A. Riggs, Shell Oil Co.

The Servomechanical Integrator in Laboratory and Process Gas Chromatography—D. W. Spence and H. L. Pope, Jr., Texas Instruments, Inc.

NUCLEAR MAGNETIC RESONANCE

B. L. Shapiro, Presiding

Nuclear Magnetic Resonance Study of Butadiene-Isoprene Copolymers—H. Y. Chen, U. S. Industrial Chemicals Co.

Carbon-13 Nuclear Magnetic Resonance. Studies of Olefins, Tertiary Butyl, and Other Compounds—R. A. Friedel and H. L. Retcofsky, U. S. Bureau of Mines

High Resolution Nuclear Magnetic Resonance of Explosives—T. Richter and H. D. Fair, Jr., Picatinny Arsenal

The Determination of Unsaturation and Average Molecular Weights of Natural Fats by NMR—L. F. Johnson and J. N. Shoolery, Varian Associates

Quantitative Applications of High Resolution Nuclear Magnetic Resonance. The Identification of Nonionic Surfactants—P. W. Flanagan, R. A. Greff, Jr., and H. F. Smith, Continental Oil Co.

Quantitative Determination by NMR of Methylene Bridge Linkages in Phenol-formaldehyde Polymers—R. C. Hirst, W. J. Burke, and D. M. Grant, University of Utah

Monday Afternoon—1:30 P. M.

ANALYTICAL CHEMISTRY—AUTOMATED METHODS

George Krapf, Presiding

Automated Fischer Retorts for Assaying Oil Shale and Bituminous Materials—A. B. Hubbard, U. S. Bureau of Mines

Automated Recording Electromagnetic Balance with Higher Capacity—L. Cahn and H. Schultz, Cahn Instrument Co.

Applications of Automatic Combustion Instrumentation—L. Malter, Coleman Instruments, Inc.

Automatic Chemical Analysis Instrumentation—R. Bull and L. Malter, Coleman Instruments, Inc.

The Use of a Specially Designed Refractometer for the Determination of Protein in a Heterogeneous Mixture—R. N. Rand, Bausch and Lomb, Inc.

Automatic Fluoride Distillation Apparatus—C. D. Herron and H. W. Thieman, Mallinckrodt Chemical Works

Multiple Liquid Stream Sampling for Automatic Potentiometric Analysis—W. J. Harrison, Fisher Scientific Co.

Quantitative Analysis with a New Thermometric Titrator—J. C. Wasilewski, American Instrument Company and J. Jordan, R. A. Javick, and M. W. Young, The Pennsylvania State University

SYMPOSIUM ON GAS CHROMATOGRAPHY—THEORY AND CAPILLARY COLUMNS

A. J. Streiff, Presiding

Resistance to Mass Transfer in the Capillary Columns—G. S. F. Hazeldean and R. P. W. Scott, Benzole Producers, Ltd.

Liquid Distribution on Gas Chromatographic Support: Relationship to Plate Height—J. C. Giddings, University of Utah

Capillary Columns for Gas-Solid Chromatography—A. Zlatkis and J. Q. Walker, University of Houston

Aluminum Capillary Columns for Gas Chromatography—D. L. Petittjean and C. J. Leftault, Jr., Aluminum Company of America

Systematic Temperature Programming Gas Chromatography—A. S. Said and T. Johns, Beckman Instruments, Inc.

Wide Range Application of Golay Columns—L. S. Ettre, The Perkin-Elmer Corp.

Design Considerations and Performance of a Linear Programmed-Temperature Gas-Liquid Chromatograph for Golay Columns—Part I, H. A. Gill—Part II, W. Averill, The Perkin-Elmer Corp.

ANALYTICAL CHEMISTRY—GENERAL

R. P. L. Hartley, Presiding

Hydrolysis Variables in the Analysis of Aluminum Alkyls—H. T. Ford and D. F. Hagen, Continental Oil Co.

Thermoanalytical Characterization by Thermogravimetry and Differential Thermal Analysis Under Controlled Conditions of Pressure and Flow in Selected Gaseous Atmospheres—S. Gordon, Fairleigh Dickinson University

Critical Angle Refractometry Applied to Continuous Liquid Stream Analysis—L. E. Maley, Waters Associates

Diffusion Method for Determination of Urinary Fluoride—R. J. Rowley, Aluminum Company of America

Determination of Iodide in Lead Telluride Semiconductor—L. Silverman, Atomics International

Tuesday Morning—8:30 A. M.

ANALYTICAL CHEMISTRY—ORGANIC ANALYSIS

M. Katz, Presiding

A New Automatic Spectrophotometric Rate Method for Selective Determination of Glucose in Serum, Plasma or Blood—H. V. Malmstadt and S. I. Hadjiioannou, University of Illinois

Specific Enzymatic Determination of Alcohol in Blood by an Automatic Spectrophotometric Reaction Rate Method—H. V. Malmstadt and T. P. Hadjiioannou, University of Illinois

Spectrophotometric Determination of Phenols, Aromatic Hydrocarbons and Hydrazine with Tetracyanoethylene—G. Schenk, M. Santiago and Patricia Wines, Wayne State University

Determination of Flexzone 3C and Other p-Phenylenediamine Derivatives in Oil Extended SBR—C. L. Hilton, United States Rubber Co.

Photometric Titration of Alkyl Aluminum Compounds—C. W. Wadelin, Goodyear Tire and Rubber Co.

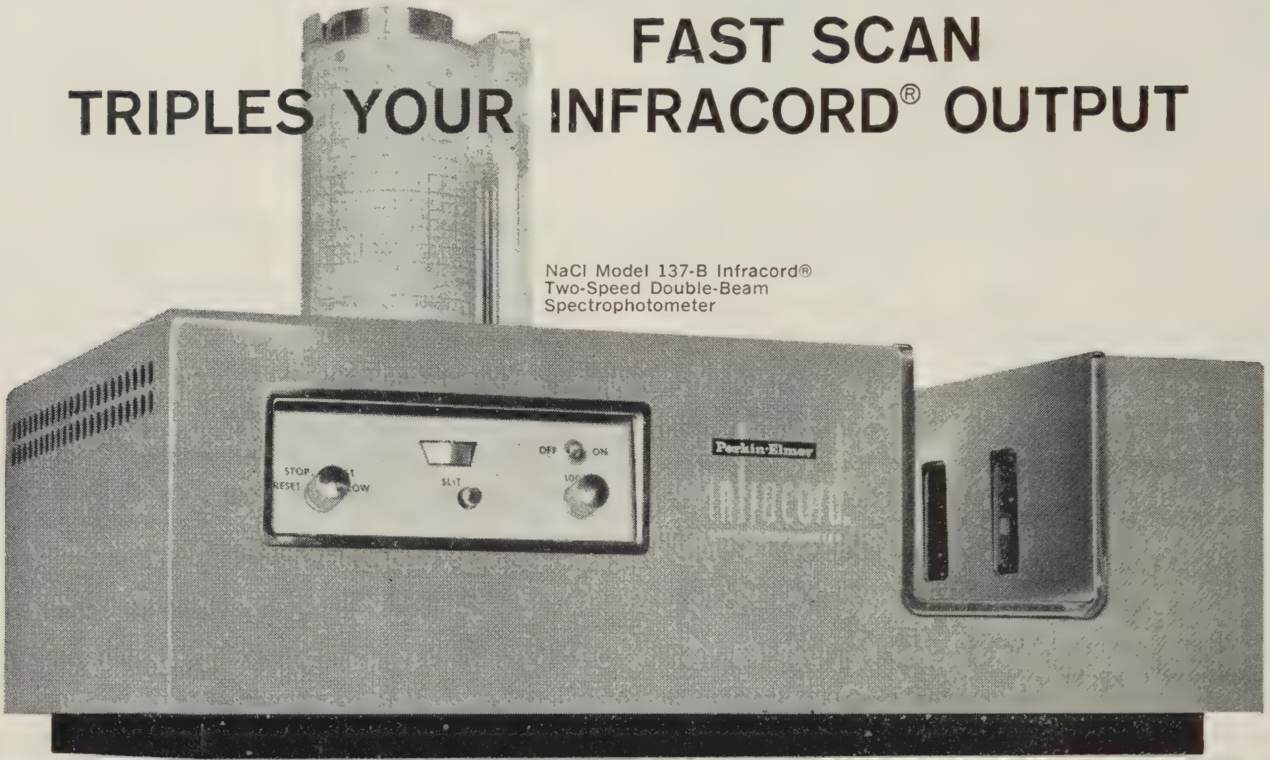
The Separation and Analysis of Azides in Organic Solvents—E. L. Grove, R. B. Bramen, H. F. Combs, and S. B. Nicholson, Armour Research Foundation

Titrimetric Determination of Phenolic Antioxidants: The Determination of 4,4′-Thiobis-(6-tert-Butyl-m-Cresol) (Santonox R)—L. P. Morgenthaler, Western Electric Engineering Research Center

Determination of Acylamidines and Other Closely Related Classes of Compounds by Non-Aqueous Potentiometric Titration—B. H. Beggs and R. D. Spencer, Mellon Institute

(Continued on page 23A)

NEW THREE-MINUTE FAST SCAN TRIPLES YOUR INFRACORD® OUTPUT



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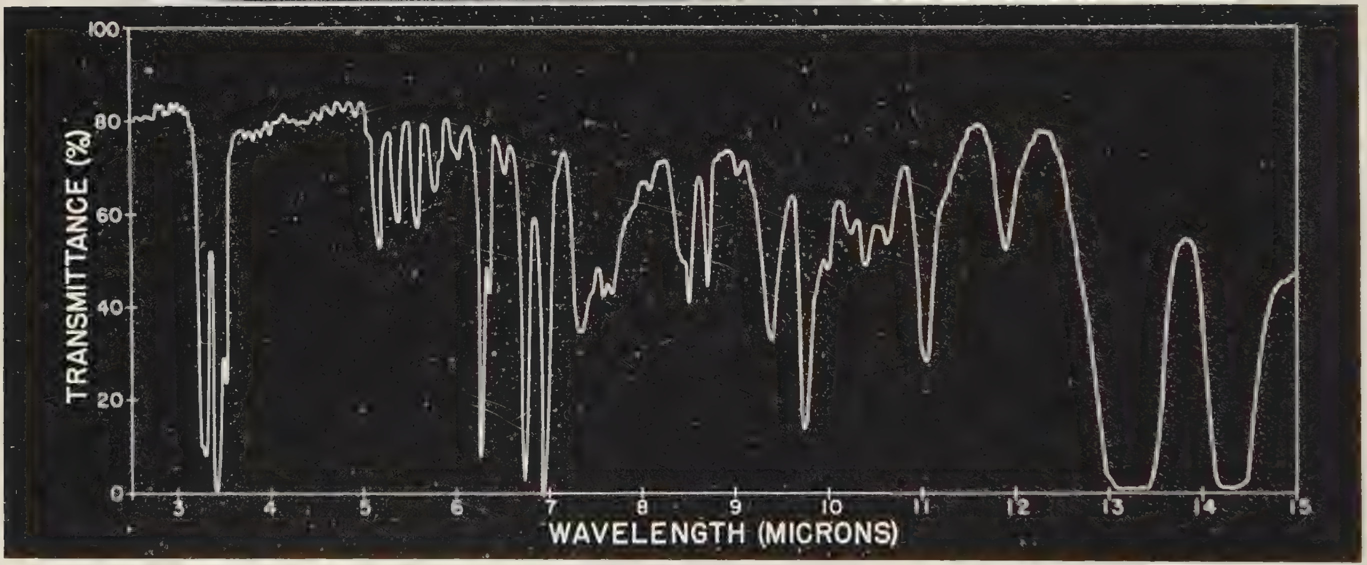

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Sample Injection Techniques and Sample System Parameters When Monitoring Liquid Chromatography Columns with a Differential Refractometer—G. E. Stackhouse, J. L. Waters and L. E. Maley, Waters Associates

GAS CHROMATOGRAPHY—ANALYSIS OF GASES

S. J. Metlin, Presiding

A Sensitive Carbon Determination by Combustion and Gas Chromatography—J. B. Mooney and L. J. Garbini, Varian Associates

Analysis of Fixed Gases, Hydrocarbons and Related Compounds by Gas Chromatography—D. M. Ottenstein, Fisher Scientific Co.

Automatic Monitoring of Atmospheres in Blast Furnaces with a New Gas Chromatograph—O. C. Karpathy and C. H. Johnson, Research and Control Instruments, Inc.

Calibration of Gas Chromatographs for Quantitative Gas Analysis—C. V. Rockoff, The Mateson Company, Inc.

Analysis of Natural Gas for Organic Sulfur Compounds by Two-Stage Gas Chromatography—B. H. Andreen and D. V. Kniebes, Institute of Gas Technology

Apparatus and "Chromatographic" Method for Studying the Adsorption of Traces of Hydrocarbons from Air—H. Fay and M. W. Voelker, Linde Company, Division of Union Carbide Corp.

Determination of Traces of CO_2 in Liquid Chlorine—C. Breton, J. E. Deutschman, P. Ede, and J. D. Zwicker, Aluminum Co. of Canada, Ltd.

Gas Chromatographic Separation of Chlorine, Hydrochloric Acid and Phosgene—W. H. Gunn and R. A. Murie, Diamond Alkali Research Center

Gas Chromatographic Analysis of Impurities in Phosgene—H. J. Hoenes, Jr., H. C. Proehl, and Z. Nagy, Pittsburgh Plate Glass Co.

NUCLEONICS

Oscar Menis, Presiding

Condensation Nuclei—A New Technique for Gas Analysis—F. W. Van Luik, Jr. and R. E. Rippers, General Electric Co.

Some Applications of Gas Detection by Condensation Nuclei Technique—F. W. Van Luik, Jr., and R. E. Rippere, General Electric Co.

Thallium-204 Radiometric Analysis for Dissolved Oxygen in Water—H. G. Richter and A. S. Gillespie, Jr., Research Triangle Institute

A Radio-Chemical Method for Measuring Radical Yields in the Radiolysis of Liquid Hydrocarbons—R. Holroyd and G. Klein, Mellon Institute

The Routine Analytical Application of Delayed-Neutron Counting to the Determination of U-235—F. F. Dyer and G. W. Leddicotte, Oak Ridge National Laboratory

Tuesday Afternoon—1:30 P. M.

ANALYTICAL CHEMISTRY—SYMPOSIUM ON CHEMICAL ANALYSIS OF METALS

Hugh Beeghly, Presiding

Recent British Developments in the Determination of Oxygen and Hydrogen in Steel—C. E. A. Shanahan, Richard Thomas & Baldwins, Ltd.

A New Technique for the Determination of the Oxygen and Nitrogen Content of Metals—V. A. Fassel and F. M. Evens, Iowa State University

Determination of Microquantities of Nitrogen in Steel—L. C. Pasztor, Jones and Laughlin Steel Corp.

A New Rapid Method for the Direct Determination of Nitrogen in Metals—F. C. Schultz, Laboratory Equipment Corp.

The Photometric Determination of Antimony in Steels—J. Penkrot, Westinghouse Electric Corp.

Determination of Trace Quantities of Sulfur in Metals—K. E. Burke and C. M. Davis, International Nickel Company, Inc.

Spectrophotometric Method for the Determination of Zirconium in Iron, Nickel, and Cobalt Base Alloys—W. J. Raber, Crucible Steel Company of America

The Polarographic Determination of Antimony in Steel—W. R. Bandi, C. C. Wareham, and L. M. Melnick, United States Steel Corp.

GAS CHROMATOGRAPHY—GENERAL

D. L. Petitjean, Presiding

Gas Chromatographic Analysis of Aqueous Organic Solutions—W. Aznavourian and E. A. McIntyre, The Foxboro Co.

The Separation of Lower Aliphatic Amines by Gas Chromatography—Y. L. Sze and M. L. Borke, Duquesne University; D. M. Ottenstein, Fisher Scientific Co.

Analysis of Formaldehyde by Gas Chromatography—K. J. Bombaugh and W. C. Bull, Spencer Chemical Co.

Aliphatic and Polycyclic Hydrocarbons in Urban Atmospheres—F. T. Fox and E. Sawicki, Robert A. Taft Sanitary Engineering Center.

Gas Chromatography of Pyrolytic Products of Purines and Pyrimidines—E. G. Jennings, Jr. and K. P. Dimick, Wilkens Instrument & Research, Inc.

Gas Chromatographic Analysis of Various Silanic Esters C. R. Thrash, Union Carbide Corp.

Quantitative Analysis of a Homologous Series of Alcohols and Hydrocarbons. Programmed Temperature Gas Chromatography with Glass Microbeads—J. G. Nikelly, Esso Research & Engineering Co.

The Study of Flame Response and Quantitative Determination of High Molecular Weight Paraffins and Alcohols—G. Perkins, Jr., L. D. Lively, and R. E. Laramy, Continental Oil Co.

Response of the Katharometer to Polar Compounds as Compared to Saturated Hydrocarbons—R. E. Laramy, L. D. Lively, and G. Perkins, Jr., Continental Oil Co.

Wednesday Morning—8:30 A. M.

ANALYTICAL CHEMISTRY—NON-FERROUS METALS

F. P. Byrne, Presiding

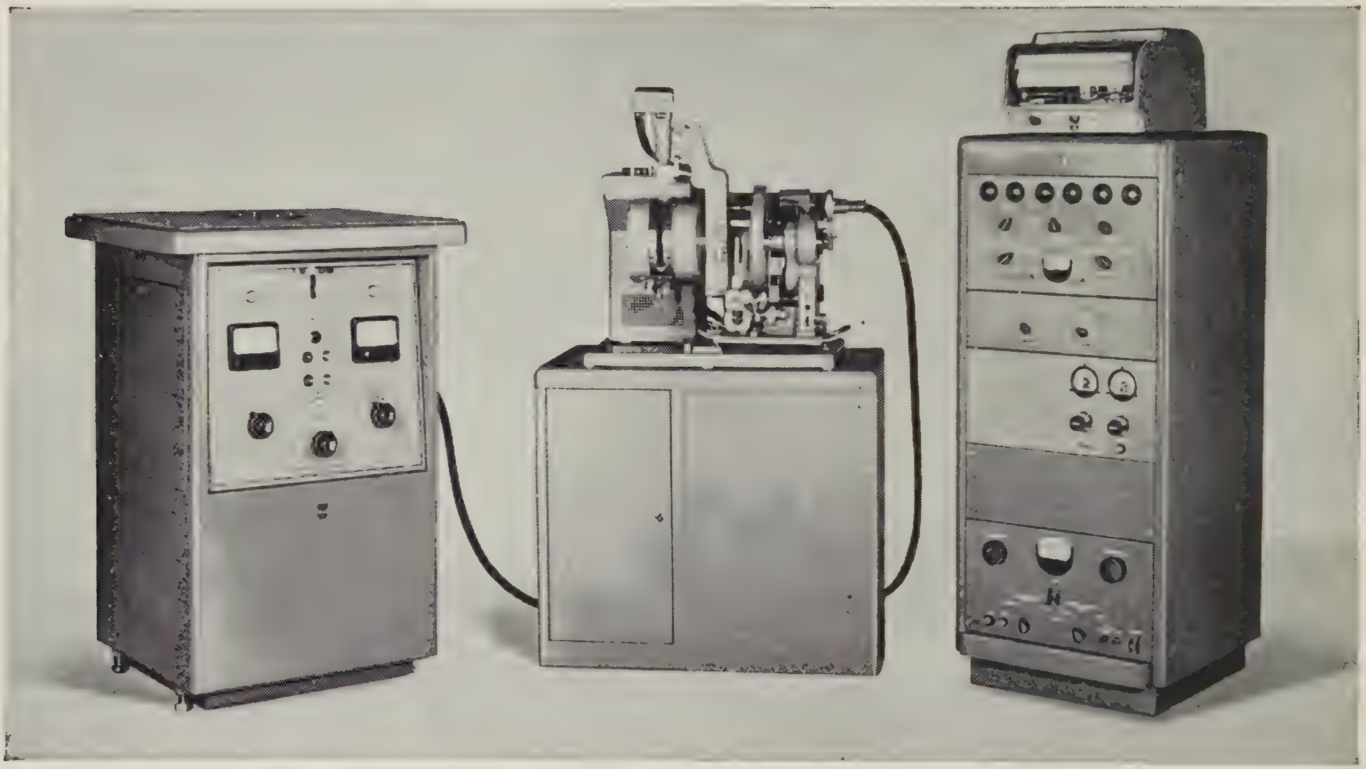
A Study of the Nickel Complex of 0,0-Dialkyl Phosphorodithioic Acids—D. Roy, Victor Chemical Works

Determination of Silver Ions in Solution with a Glass Electrode—A. L. Budd, Beckman Instruments, Inc.

(Continued on page 25A)

NEW NON-DESTRUCTIVE CHEMICAL ANALYSIS

PARTS PER MILLION TRACINGS—WITH FANTASTIC ACCURACY AND SPEED



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Rapid Direct EDTA Determination of Aluminum and Magnesium in Zinc Base Die Casting Alloys—C. M. Smink and R. G. Mercer, New Jersey Zinc Co.

The Conductometric Determination of Carbon Below 10 ppm in Zone Refined Tungsten—W. A. Gordon, Z. T. Tumney, and J. W. Graab, National Aeronautics and Space Administration

SYMPOSIUM COMMEMORATING THE FIFTEENTH ANNIVERSARY OF THE FISHER AWARD IN ANALYTICAL CHEMISTRY

L. M. Melnick, Moderator

Analytical Chemistry Clinic

The Fisher awardees will be a panel to which questions can be directed from the floor on problems of analyses or general topics relating to analytical chemistry.

10:15 a.m.—Opening remarks by the Conference General Chairman, Fritz Will, III.

Awardees participating; N. H. Furman, I. M. Kolthoff, H. H. Willard, M. G. Mellon, G. F. Smith, E. H. Swift, H. C. Diehl, J. H. Yoe, J. J. Lingane, J. I. Hoffman, P. J. Elving, and H. A. Liebhasky.

GAS CHROMATOGRAPHY—GENERAL

D. H. Lichtenfels, Presiding

Prediction of Retention Times—H. J. Maier, O. C. Karpathy, and N. Brenner, The Perkin-Elmer Corp.

The Characterization of High Molecular Weight Substances by Gas Chromatography—L. Mikkelsen and M. G. Beck, F & M Scientific Corp.

Gas Chromatography in Chemical Research—W. W. Brandt, Kansas State University.

Wednesday Afternoon—1:00 P. M.

X-RAY SPECTROSCOPY—INSTRUMENTATION AND APPLICATIONS

R. K. Scott, Presiding

A Practical Approach to the Analysis of Iron Ores Using X-Ray Quantometers—H. T. Dryer and B. R. Boyd, Applied Research Laboratories, Inc.

X-Ray Spectroscopic Analysis of Noble Metal Alloys—B. W. Mulligan, H. J. Caul, S. D. Rasberry, and B. F. Scribner, National Bureau of Standards

A Semi-Quantitative X-Ray Fluorescence Analysis System—K. F. J. Heinrich, E. I. du Pont de Nemours and Co., Inc.

X-Ray Fluorescence Methods for the Determination of Tin Plate Coating Weights—J. H. Kelly and D. E. Cass, The Steel Company of Canada, Ltd.

Analysis of Solutions by X-Ray Spectroscopy—T. J. Cullen, United States Metals Refining Co.

An Improved X-Ray Fluorescence Method for Trace Metals in Refinery FCC Feedstocks—W. A. Rowe and K. P. Yates, The Pure Oil Co.

Detector Adjustment Technique for Minimizing Errors in X-Ray Emission Spectroscopy—D. D. Grewe, General Electric X-Ray Department.

Use of the Electron Beam Microanalyzer for the Identification of Stratospheric Particles—Susan Schur, Advanced Metals Research Corp.

Performance Evaluation of the Electron Microprobe X-Ray Analyzer—E. Davidson, W. E. Fowler, and H. Neuhaus, Applied Research Laboratories, Inc.

Design Considerations for a New Vacuum Autrometer—W. R. Kiley, Philips Electronic Instruments.

EMISSION SPECTROSCOPY—SYMPOSIUM ON VACUUM ULTRAVIOLET SPECTROSCOPY ORGANIZED BY THE SOCIETY FOR APPLIED SPECTROSCOPY

J. E. Paterson, Presiding

SPECTROSCOPY SOCIETY OF PITTSBURGH 1962 AWARD ADDRESS

2:00 p.m.—Presentation of 1962 SSP Award by D. L. Petitjean, Chairman, Spectroscopy Society of Pittsburgh.

Award address by Dr. G. Herzberg, National Research Council, Canada, *Molecular Spectra in the Vacuum Ultraviolet.*

Absorption Spectroscopy in the Extreme Ultraviolet—J. F. Rendina, E. I. duPont de Nemours and Company, Inc.

Spectrographs and Monochromators for Vacuum Ultraviolet Spectroscopy—T. Namioka, Air Force Cambridge Research Laboratories

Photoelectric Detection and Spectral Discrimination of Extreme Ultraviolet Radiation—H. E. Hinteregger, Air Force Cambridge Research Laboratories

Recent Advances in the Study of Reflecting Films for the Vacuum Ultraviolet—R. P. Madden, National Bureau of Standards

SYMPOSIUM ON THE SPECTROSCOPIC STUDY OF POLYMERS ORGANIZED WITH THE COBLENTZ SOCIETY

A. W. Baker, Presiding

Infrared Spectra of Irradiated Polyolefins—J. P. Luongo and R. Salovey, Bell Telephone Laboratories, Inc.

Infrared Studies of Alkyd and Monomer-Modified Alkyd Resins—R. L. Harris and G. Svoboda, Freeman Chemical Corp.

A Kinetic Method for the Determination of Phenol-Formaldehyde Ratios in Phenolic Resins by Infrared Spectroscopy—R. T. Conley and J. F. Bieron, Seton Hall University

A Study of the Oxidative Degradation of Condensation Polymers Using Infrared Spectroscopy—R. T. Conley, Seton Hall University

Some Changes in Double Bond Structure During the Vulcanization of Natural Rubber—F. J. Linnig, National Bureau of Standards and J. E. Stewart, Beckman Instruments, Inc.

Thursday Morning—8:30 A. M.

MOLECULAR SPECTROSCOPY—INSTRUMENTATION

D. S. McKinney, Presiding

Design and Construction of An Infrared Polychromator—Y. Mashiko and H. Tomita, The Government Chemical Industrial Research Institute of Tokyo and K. Yoshida, Hitachi, Ltd.

New Filter-Grating Infrared Spectrophotometer—G. T. Keahl, Beckman Instruments, Inc.

(Continued on page 27A)

NO. 77 NMR AT WORK SERIES

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NMR SPECTROSCOPY IS NOW A QUANTITATIVE ANALYTICAL TECHNIQUE

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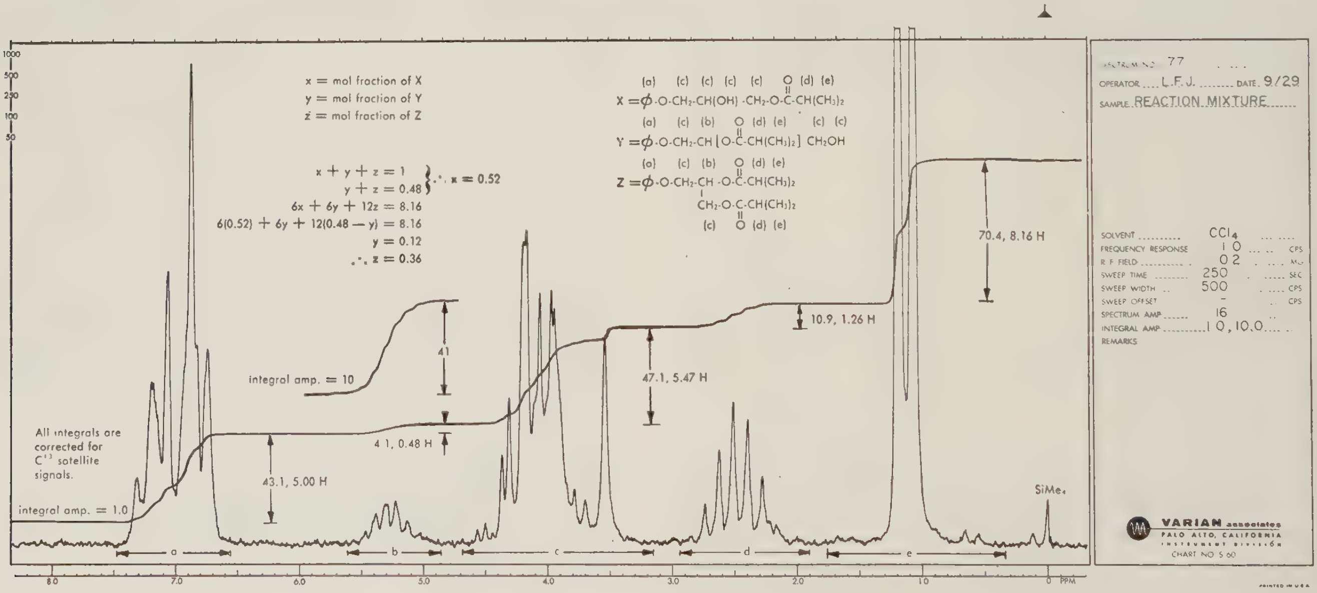
EXAMPLE: *Reaction mixture analysis*

The reaction between phenyl glycidyl ether and isobutyric acid produces a mixture of three esters.(1) NMR signals from these components, whose structures are shown on the trace below, fall into five well defined areas in the spectrum, labeled (a) through (e). Proton groups in each compound producing these signals are labeled correspondingly.

Since each component structure contains five phenyl protons, the integral per proton is calibrated using area (a). Components Y and Z each have a single proton which contributes to area (b); therefore, the sum of their mol fractions and consequently the mol fraction of the remaining component X are readily calculated. Y and Z, having a different number of methyl groups, can be determined using area (e). The results can be internally checked by calculating the total number of hydrogens, 18(0.52) + 18(0.12) + 24(0.36) = 20.2, and comparing with the sum of protons in each area, 5.00 + 0.48 + 5.47 + 1.26 + 8.16 = 20.4.

The analysis is completed without the use of the pure components since the chemical shifts are readily predicted with sufficient accuracy in acylic compounds, and the final results are self-consistent.

(1) *Permission to describe this research problem at Thiokol Chemical Corp. is appreciated.*



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The Model 125 Grating Spectrophotometer—A High Performance Instrument for the Range 10,000-400 cm^{-1}—V. J. Coates and H. M. Bolz, Perkin-Elmer Corp.

A New Spectrophotometer—J. M. Thoburn, Bausch and Lomb, Inc.

A New Extended Range Infrared Spectrophotometer—J. Ashley and N. Shifrin, Beckman Instruments, Inc.

An Instrument for Simultaneous Integration of Concentration of Each Infrared Absorbing Gas in a Multi-component Flowing Stream—G. Smith and P. Watson, Baird-Atomic, Inc.

A Compact High Intensity Monochromator—J. J. Chisholm, Bausch and Lomb, Inc.

New Instrumentation for Fluorescence and Phosphorescence Analysis—J. U. White, White Development Corp., and R. Ploss and J. Leeman, Baird-Atomic, Inc.

A Fluorescence Attachment for Beckman DU and DK Spectrophotometers—M. Laikin, Beckman Instruments, Inc. and J. F. Thomas, Sanitary Engineering Laboratory, University of California Field Station

An Attachment for Spectrophotometry of Scattering and Fluorescing Samples—R. Eno and R. C. Hawes, Applied Physics Corp.

EMISSION SPECTROSCOPY—INSTRUMENTATION

J. D. Johnson, Presiding

A New Emulsion for Spectrochemical Analysis—R. L. Dehm, Eastman Kodak Company.

The Jarrell-Ash Vacuum Atomcounter—R. W. Tabeling, J. Devaney, and F. Brech, Jarrell-Ash Co.

Design of a New Direct Reading Spectrometer—E. Dubois, Research and Control Instruments, Inc.

Spectrochemical Analysis in the Near Schumann Region—A. L. Davison, Baird Atomic, Inc.

Two Coordinate Photoelectric Setting Attachment Device for Precision Measuring Comparator—L. O. Eikrem and H. B. Beal, David W. Mann Company, Division of Geophysics Corporation of America

A New Atomic Absorption Photometer—J. L. Saunderson, Research and Control Instruments, Inc.

High Sensitivity Approaches to Atomic Absorption—J. Devaney and F. Brech, Jarrell-Ash Co.

SYMPOSIUM ON THE SPECTROSCOPIC STUDY OF POLYMERS ORGANIZED WITH THE COBLENTZ SOCIETY

S. Krimm, Presiding

Introductory Remarks, S. Krimm

Infrared Spectra and Structure of Polyvinyl Chloride—T. Shimanouchi, University of Tokyo

The Use of Model Compounds in Interpreting Infrared Spectra of Chlorine Containing Organic Compounds and Polymers—J. J. Shipman and V. L. Folt, B. F. Goodrich Company, and S. Krimm, University of Michigan

Infrared Spectra and Assignments for Polyvinyl Chloride and Some of Its Deuterated Analogs—S. Krimm, University of Michigan and V. L. Folt, J. J. Shipman, and A. R. Berens, B. F. Goodrich Co.

Polyethylene Terephthalate Structural Studies—P. G. Schmidt, E. I. du Pont de Nemours & Company, Inc.

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Applications of Infrared Spectroscopy to the Study of Wood Polysaccharides—R. H. Marchessault, Syracuse University

Thursday Afternoon—1:30 P. M.

EMISSION SPECTROSCOPY—ATOMIC ABSORPTION, FLAME AND GAS ANALYSIS

E. R. Shuster, Presiding

Performance Characteristics of Hollow Cathode Gas Discharge Devices for Atomic Absorption Spectroscopy—J. C. Burger and W. Gillies, Westinghouse Electric Corp.

The Elimination of Interferences in Atomic Absorption Spectrochemical Analysis—D. J. David, Division of Plant Industry, Australian Commonwealth Scientific and Industrial Research Organization

Elimination of Calcium Interference in the Flame Photometric Determination of Sodium and Potassium in Calcium Salts—J. W. Hunter, General Electric Co.

New Sources for Flame Spectrophotometry—P. T. Gilbert, Jr., Beckman Instruments, Inc.

A Spectroscopic Method and Analyzer for Traces of Nitrogen in Argon—H. Fay and P. H. Mohr, Linde Company, Division of Union Carbide Corp.

MOLECULAR SPECTROSCOPY—GENERAL

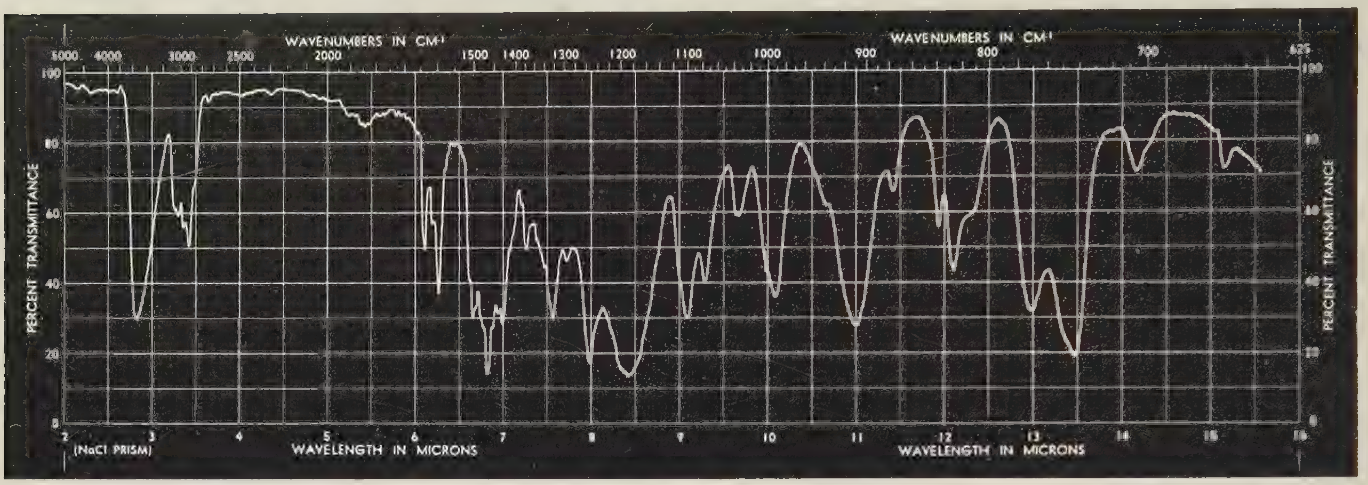
M. S. Norris, Presiding

A Raman Spectroscopic Study of the Structures of Some Solid Complexes of BF_3 and the Nitrogen Oxides—J. C. Evans, S. J. Kuhn, and G. A. Olah, Dow Chemical Co.

General Basis for Quantitative Raman Analysis—D. D. Tunnicliff and A. C. Jones, Shell Development Co.

(Continued on page 29A)

WHAT IS IT ?...



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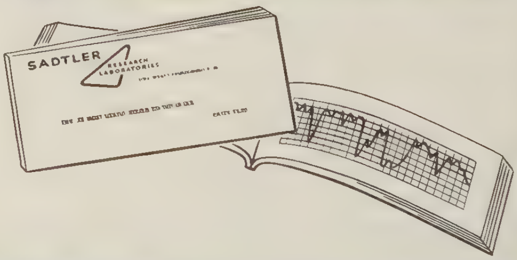

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A Multiple-Pass Raman Cell—D. D. Tunnicliff and A. C. Jones, Shell Development Co.

Some Useful Applications in Ultraviolet Spectroscopy—T. J. Porro, The Perkin-Elmer Corp.

Evidences of Hydrogen Bonding in Far Ultraviolet Spectra—W. Kaye and R. Poulson, Beckman Instruments, Inc.

Improving Spectrophotometric Measurements in the "Near Vacuum" Ultraviolet—R. C. Hawes, E. R. Schweitzer, R. E. Ellis, and W. R. Williams, Applied Physics Corp.

Electronic Emission Spectra of Some Mono-, Di-, and Tri-Substituted Quinolines—H. K. Howerton, American Instrument Company, Inc., and J. M. Price, University of Wisconsin

Analytical Reagents for Fluorescence Spectroscopy—H. K. Howerton, American Instrument Company, Inc.

A Theoretical Treatment of the Causes of Curvature of Fluorimetric Calibration Curves—D. D. Harmon, Bausch and Lomb, Inc.

Automatic Spectral Scanning of Large Numbers of Samples of Widely Different Absorbances—J. G. Erdman, C. L. Wolfe, and G. A. Sill, Mellon Institute

An Instrumental Aid for the Interpretation of Spectral or Other Chart Recorded Data—J. G. Erdman, C. L. Wolfe, and W. C. Barnes, Mellon Institute

SYMPOSIUM ON THE SPECTROSCOPIC STUDY OF POLYMERS ORGANIZED WITH THE COBLENTZ SOCIETY

S. Krimm, Presiding

Infrared Spectrum of Polypropylene—C. Y. Liang, American Viscose Corp.

Vibrational Spectra of Crystals of Compounds with Long Methylene Chains—J. R. Nielsen, R. F. Holland and C. E. Hathaway, University of Oklahoma

Intramolecular Force Field for Polyethylene—R. G. Snyder, Shell Development Co.

Complete Vibrational Analysis of the Isolated Polyethylene Chain—T. P. Lin and J. L. Koenig, E. I. du Pont de Nemours and Company, Inc.

Friday Morning—8:30 A. M.

EMISSION SPECTROSCOPY—CONTROLLED ATMOSPHERES AND TRACE ANALYSIS

R. F. Farrell, Presiding

Performance of Controlled Atmosphere Chamber—D. O. Landon and A. J. Mitteldorf, Spex Industries, Inc.

Spectrochemical Determination of Microgram and Submicrogram Amounts of Beryllium in Air Dust—R. L. O'Neil, A. C. Spark Plug Division, General Motors Corp.

The Influence of Certain Gases on the D. C. Arc, Using the Stallwood Jet—J. T. Peters and M. Lalevic, Drexel Institute of Technology

The Controlled Atmosphere D. C. Arc for the Quantitative Determination of Trace Metals in Organics—D. L. Nash, Bell Telephone Laboratories, Inc.

Spectrochemical Studies of the Ge-Ga System—W. Reuter and J. C. Lloyd, IBM Corp.

The Determination of Alloying Constituents in Semiconductor Dot Alloys Using a Dot to Dot Technique—W. E. Sonia, Jr. and J. M. Dendy, Texas Instruments, Inc.

Direct Spectrographic Determination of Trace Impurities in High Purity Platinum—A. J. Lincoln and J. Kohler, Engelhard Industries, Inc.

Spectrographic Determination of Trace Impurities in High Purity Gold—A. J. Lincoln and J. Kohler, Engelhard Industries, Inc.

INFRARED SPECTROSCOPY—GENERAL

Foil A. Miller, Presiding

A Compilation and Analysis of the Band Positions of a Number of Compounds—H. A. Szymanski, Canisius College

Infrared Spectra of Several Salts of Acidic Organophosphorus Compounds—J. R. Ferraro, Argonne National Laboratory

Inductive Effects in the Infrared Spectra of Substituted Silanes—A. L. Smith and N. C. Angelotti, Dow Corning Corp.

Infrared Correlations of Compounds Containing the Group $>\overset{\|}{\mathrm{P}}\text{-NH}_2$ or $>\overset{\|}{\mathrm{P}}\text{-NHR}$ (Frequencies, Intensities, and Band Shapes)—R. A. Nyquist, Dow Chemical Co.

The Effects of Polar Groups on Alkane C-H Stretching and Bending Frequencies—W. J. Potts, Dow Chemical Co.

Infrared Absorption Patterns of Disubstituted Naphthalenes—D. W. Heinritz and W. M. Padgett, II, Hyman Laboratories, Inc.

The Influence of External Halide Ions on the Infrared Spectrum of the p-Chloroanilinium Ion—Sister Miriam Michael Stimson, O.P., and Linda L. Smith, Siena Heights College

Infrared Spectroscopic Study of Aliphatic Alcohol-Ester Hydrogen Bonding—A. S. Rosenberg and H. F. Smith, Continental Oil Co.

Infrared Study of Linear Acyclic Terpenes I—B. M. Mitzner and S. K. Freeman, International Flavors and Fragrances, Inc.

Infrared Study of Cyclic Terpenes II—B. M. Mitzner and S. K. Freeman, International Flavors and Fragrances, Inc.

Friday Afternoon—1:30 P. M.

EMISSION SPECTROSCOPY—STANDARDS, ANALYSIS OF METALS AND SOLUTIONS

B. M. LaRue, Presiding

Quantometric Analysis of Silicon Using a Briquetting Technique—A. D. Behel, Sr. and J. W. Pendley, Reynolds Metals Co.

The Spectrochemical Analysis of Copper Base Alloys Using a Photoelectric Spectrometer—J. T. Herlihy and G. J. Deffenbaugh, Sandusky Foundry and Machine Co.

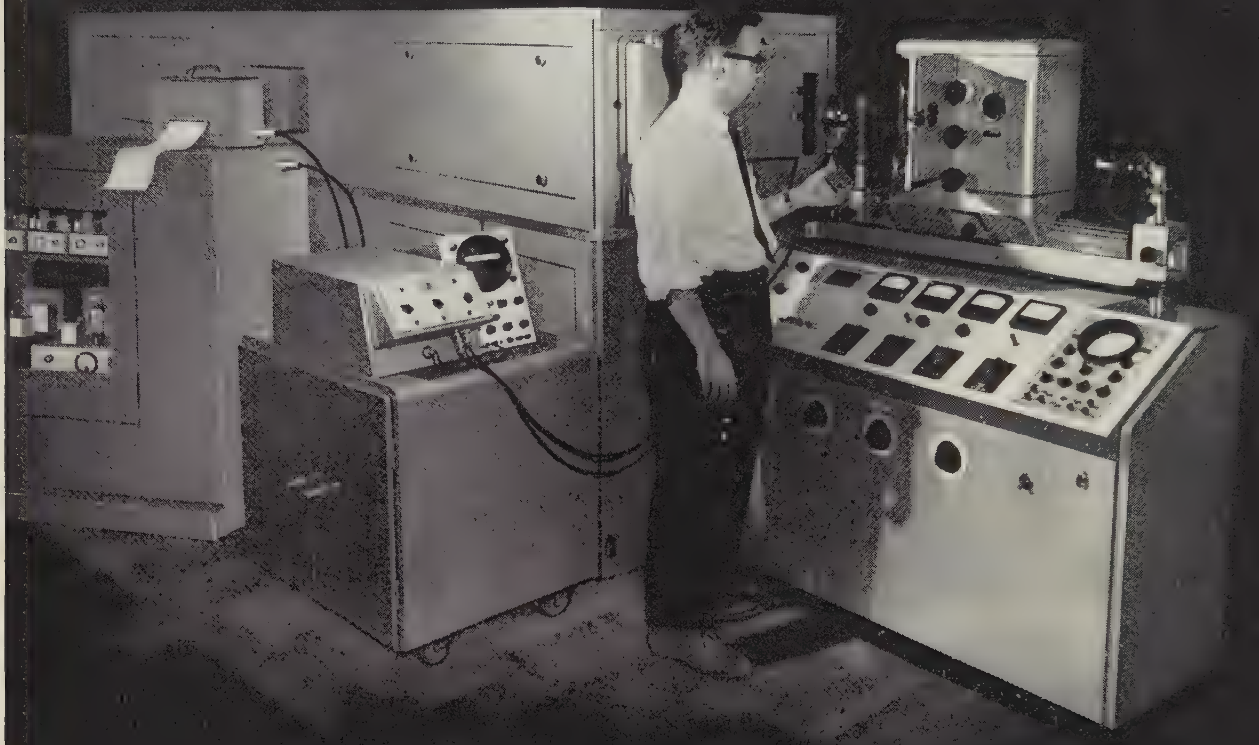
Preparation and Evaluation of NBS Copper-Base Spectroscopic Standards—R. E. Michaelis and R. Flitsch, National Bureau of Standards

Emission Spectrometric Analysis for Lead in Rolled Resulfurized Leaded Steel—J. K. Hurwitz and M. D. Smith, U. S. Steel Corp.

(Continued on page 31A)

Wyman-Gordon depends on B/A Spectrograph

to analyze wide variety of metals for aircraft and missile forgings



Wyman-Gordon Company produces a larger volume and wider variety of aircraft and missile forgings than any other metal fabricator in the world. Products include: fuselage frames (aluminum); landing wheels (magnesium); piston engine crankshafts (steel); turbine wheels (nickel-base alloy); missile re-entry shields (copper); and missile cones (titanium).

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A Filter Electrode Technique for Spectrochemical Analysis—C. L. Chaney and M. J. Peterson, U. S. Bureau of Mines

Spectrographic Analysis of Molybdenum, Molybdic Oxide, and Ammonium Paramolybdate by the Insoluble Layer Technique—J. E. Wagoner and W. J. Tschudi, Climax Molybdenum Co.

Emission Spectrographic Method for the Determination of Microgram Quantities of Ba, Sr, Ca, Mg, Mn, Si, and Ni in Sublimed Deposits on Micas, Grids, Plates, and Glass Bulbs of Electron Tubes—A. M. Liebman, Radio Corporation of America

Use of Vacuum Cup Electrodes for Multiple Excitation with a High Voltage Spark Source—S. T. Bass and J. Soulati, Michigan State University

INFRARED SPECTROSCOPY—GENERAL

R. A. Friedel, Presiding

Grating Infrared Spectroscopy in the Analytical Laboratory—R. C. Gore, The Perkin-Elmer Corp.

Frequency Accuracy of the Beckman IR-7 Infrared Spectrophotometer—P. J. Krueger, University of Alberta

An Infrared Differential Technique Employing Membrane Filters—H. J. Sloane, Beckman Instruments, Inc.

Infrared Analysis by Attenuated Total Reflectance—P. A. Wilks, Jr., Connecticut Instrument Corp.

Determination of Primary and Secondary Aliphatic Amines by Near-Infrared Spectrophotometry—W. E. Norteman and F. H. Lohman, The Procter and Gamble Co.

Infrared Method for Determination of Alkylbenzene Sulfonate Concentration in Aqueous Solution—S. D. Kullbom and H. F. Smith, Continental Oil Co.

The Effects of Aging and Pressure on the Transmission of Infrared Radiation by KBr Pellets—C. Sass, Delaware State College

A Simple, Inexpensive Heated Infrared Gas Cell—E. A. Burns, Stanford Research Institute

St. Louis Section

Date: January 11, 1962
Time: 8:00 p.m.
Place: Garavelli's Restaurant, St. Louis, Mo.
Speaker: Paul T. Gilbert, Beckman Instruments, Inc.
Topic: "Advances in Flame Photometry"

Southeastern Section

The Tenth Annual Southeastern Seminar on Spectroscopy at the University of Florida on January 10, 11, 12, 1962.

It will be sponsored by Department of Mechanical Engineering, Engineering and Industrial Experiment Station, College of Engineering, and The Southeastern Association of Spectrographers. Emphasis will be on basic fundamentals, source parameters, precision, and allied topics. The Southeastern Association of Spectrographers will also hold its annual spring meeting in conjunction with the Seminar. Complete programs will be mailed early in December. For further information write to: Professor Wm. T. Tiffin, College of Engineering, University of Florida, Gainesville, Florida.

Baltimore-Washington Section

Date: January 9, 1962
Place: W. R. Grace & Co., Clarksville, Md.
Program: A Meeting-in-Miniature
Chairman: Mr. Franklin Winter, Koppers, Inc.

Cleveland Section

Date: January 31, 1962
Speaker: Dr. Ernest Yeager, Western Reserve University
Topic: "Radio Frequency Techniques for Exciting Spectra"

Ernest Yeager Award in Spectroscopy

The Cleveland Section of the Society for Applied Spectroscopy is sponsoring the Ernest B. Yeager Award in Spectroscopy. The award consists of a certificate plus $100.00. To be eligible a student must be an undergraduate in one of the following colleges or universities: Akron University, Baldwin Wallace College, Case Institute of Technology, Fenn College, Hiram College, John Carroll University, Kent State University, Lake Erie College, Oberlin College, Western Reserve University, Wooster College, and Youngstown University. The student must have demonstrated ability in some phase of spectroscopy through course, project or honors work. Applications of spectroscopy methods in astronomy, biology, metallurgy, chemistry, geology and physics are acceptable. The award will be presented at the Annual Conference on Spectroscopy each year in May by the Cleveland Section of SAS.

For further information write to John A. Wyman, Chairman—Educational Committee, c/o The Permold Company, Post Office Box 70, Medina, Ohio.

Delaware Valley Section

Date: January 23, 1962
Place: Town House, Media, Pa.
Speaker: Dr. B. P. Stoicheff, National Research Council, Ottawa, Canada
Topic: "Application of Lasers to Raman Spectroscopy"

New England Section

The New England Section will hold all scheduled monthly meetings on the second Monday of each month at 8:00 p.m. at Devlin Hall, Boston College, Chestnut Hill, Massachusetts.

New York Section

Date: January 9, 1962
Speaker: Dr. Herbert Friedman, U. S. Naval Research Laboratory
Topic: "X-Rays From the Sun"

The New York Section will hold all monthly meetings at the Statler Hilton Hotel, New York City, New York, at 8:00 p.m., with the exception of the May 8, 1962 meeting which will be a joint meeting with the Delaware Valley Section, held at the Princeton Inn, Princeton, N. J.

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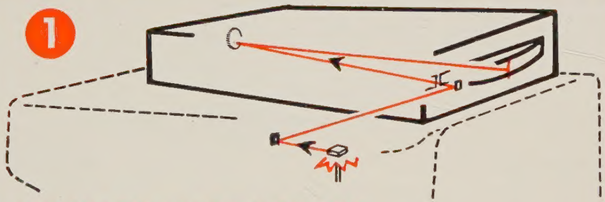

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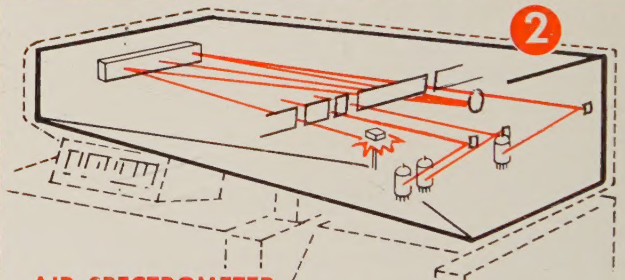

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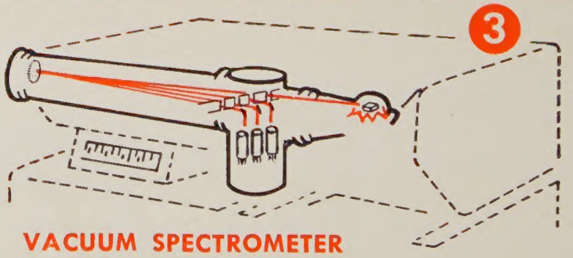

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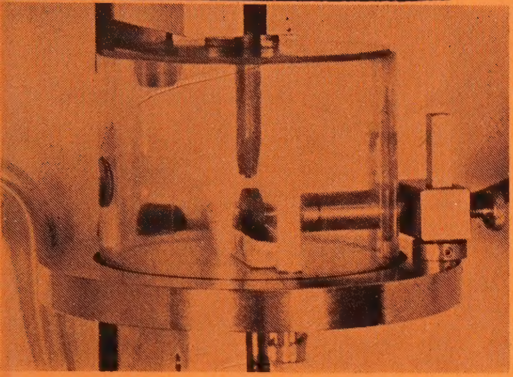

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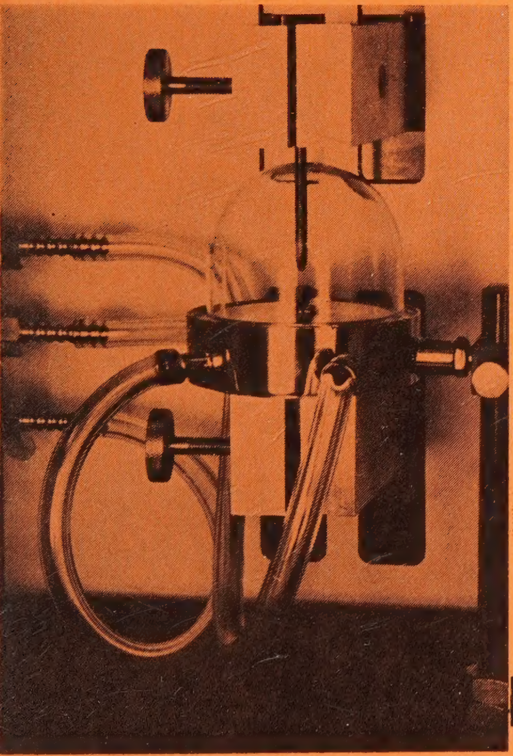
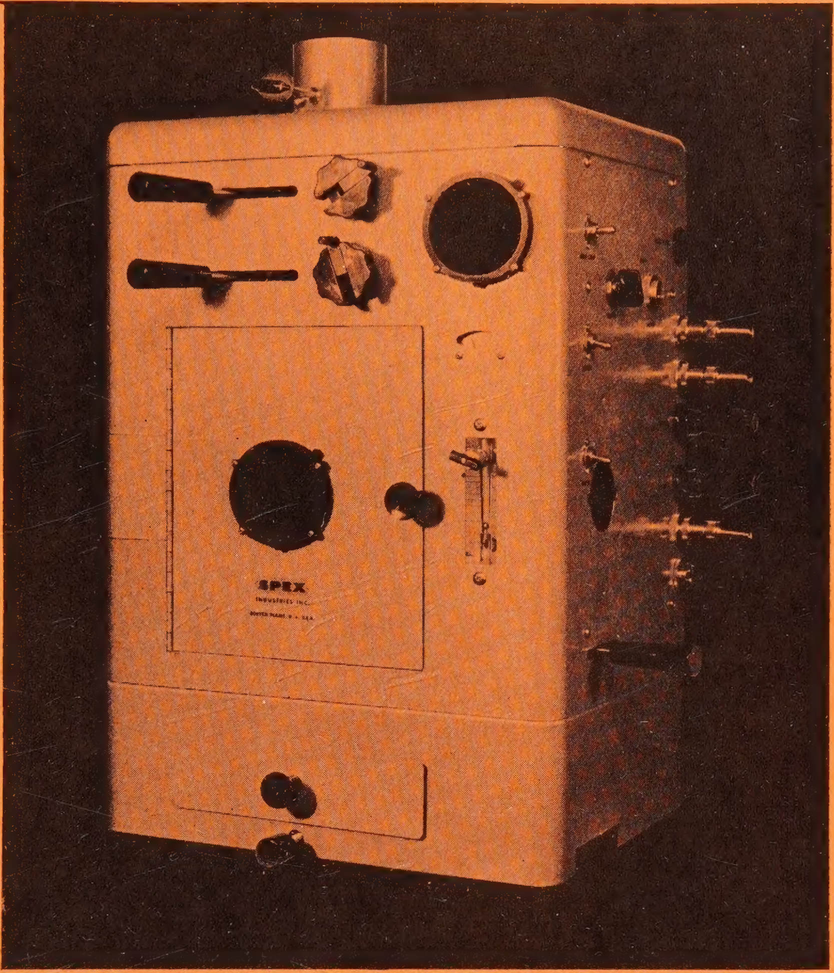


ENCLOSED STALLWOOD JET



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